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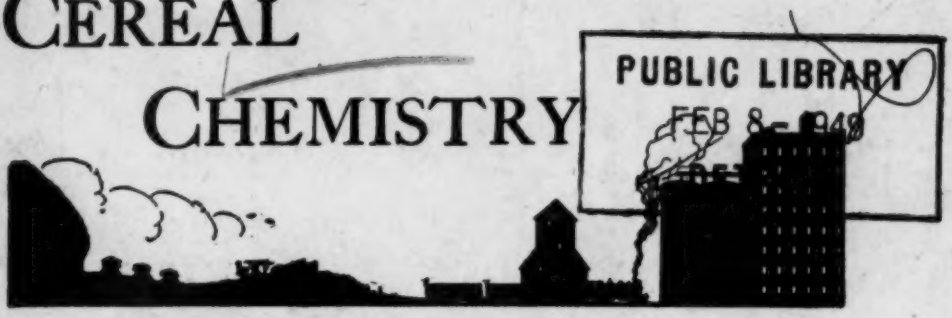
CEREAL CHEMISTRY



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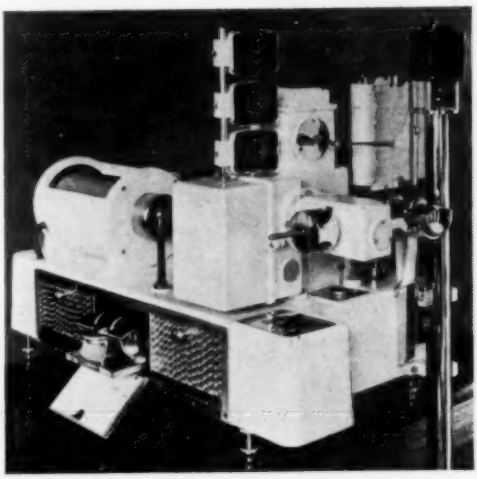

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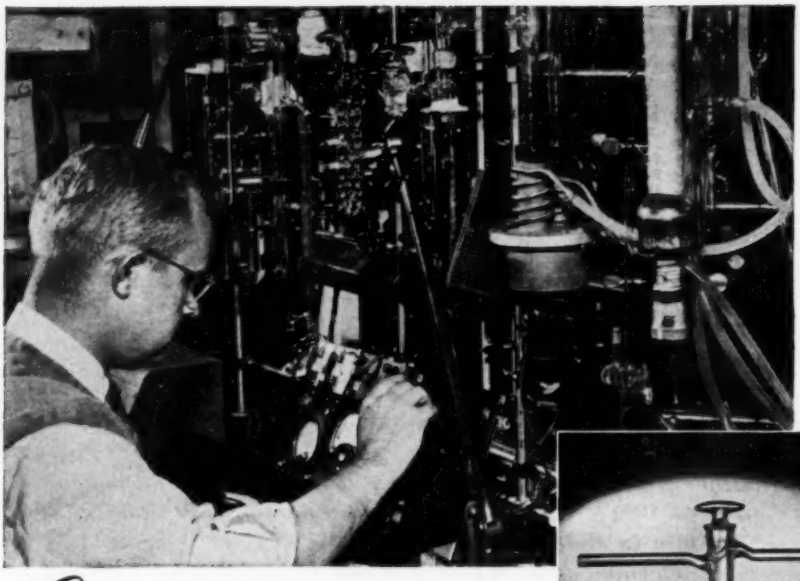

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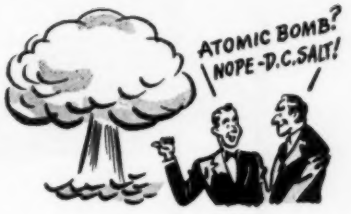

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CEREAL CHEMISTRY

VOL. XXVI JANUARY, 1949 NO. 1

HYGROSCOPIC EQUILIBRIUM OF RICE AND RICE FRACTIONS[1]

M. L. KARON and MABELLE E. ADAMS[2]

ABSTRACT

An investigation has been made of the rate of sorption and desorption of moisture by rough rice, head rice, bran, polish, and hulls over the range of 11 to 93% relative humidity at 25°C. The hygroscopic equilibrium of these same fractions has been determined over the same relative humidity range. If the relative humidity of the atmosphere at 25°C. is raised from 10 to 90% the moisture content of whole rice and its fractions increases as follows: rough rice from 4.4 to 17.6%, polished rice from 5.2 to 18.8%, bran from 5.0 to 18.0%, polish from 5.3 to 18.0%, and hulls from 3.7 to 15.3%.

In the past 15 years the combine has been increasingly used for harvesting rice. In 1947 more than one-half of the rice crop in Louisiana was harvested by combines (4). This was true of practically all of the other rice-producing areas. Rice when harvested by combine generally has a moisture content of 20% or more and must be artificially dried to 14% moisture to prevent heating and spoilage during storage. This drying operation must be carried out slowly and at relatively low temperatures. Otherwise the rice kernel loses its vitreous character and a large number of kernels will be broken during processing.

Since the moisture content of rice and rice by-products will vary with the atmospheric humidity conditions, and inasmuch as the moisture content seriously affects the storage and processing characteristics of the grain, an investigation was therefore undertaken of the hygroscopic equilibrium of rough rice, head rice, bran, polish, and hulls. Data were obtained on the rates of sorption and desorption of moisture by whole rice and its various component fractions, and the moisture content in equilibrium with air of constant relative humidity over the range of 11 to 93% relative humidity at 25°C. was determined.

[1] Manuscript received June 25, 1948. Report of a study in which certain phases were carried on under the Research and Marketing Act of 1946.

[2] Southern Regional Research Laboratory, New Orleans, Louisiana. One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

Materials

The rice used in this investigation was from two separate lots. The first, henceforth to be known as artificially dried rice, was obtained at a rice mill from a mixed lot containing 17.8% moisture which had been dried in a commercial rice drier to a moisture content of 12.8%. This dried rice was processed only to an undermilled product. The second lot, henceforth to be known as naturally dried rice, was of Rexora variety and contained 16.8% moisture as received at the mill where it was immediately processed to a finished polished rice.

Because of the extreme difficulty of quantitatively separating rough rice in the laboratory into head rice, bran, polish, and hulls without bringing about appreciable changes in moisture content, the investigations were conducted on rough rice and the rice fractions obtained from the same lot as it was being processed in a commercial mill. Samples from each milling stream were collected simultaneously, placed in sealed bottles, and taken to the laboratory. Milling data on the entire lot of the artificially dried rice indicated that one barrel (162 pounds) yielded the following products: undermilled head rice, 125.31 pounds (77.35%); bran, 7.80 pounds (4.81%); and hulls and waste, 28.89 pounds (17.83%). In the case of naturally dried rice, the yield from one barrel was as follows: head rice, second heads, and screenings, 105.87 pounds (65.35%); bran, 21.64 pounds (13.35%); polish, 1.35 pounds (0.83%); and hulls and waste, 33.14 pounds (20.46%).

Methods

Saturated solutions of pure salts were used to provide the desired series of relative atmospheric humidities. Spencer (5) lists a number of saturated salt solutions which maintain constant relative humidities over the entire range. Others may be found in the report of the American Paper and Pulp Association (1). The salts used in this investigation and the values of the corresponding relative humidities for their saturated solutions at 25°C. are listed in Table I.

Saturated salt solutions, together with an excess of solid salt, were placed in large desiccators, and samples of the rough rice, head rice, bran, polish, and hulls were suspended above these solutions by means of partitioned wire mats. The desiccators were stored in a room maintained at 25°C. Every few days the desiccators were opened and 5–10 g. duplicate samples of each fraction were removed and weighed immediately in closed moisture dishes. In all moisture determinations the products were heated at 101°C. for 16 hours in a forced-draft oven, reweighed, and the loss in weight calculated as moisture content on the wet basis.

Because the samples were in intermediary stages approaching hygroscopic equilibrium, it was not feasible to apply the vacuum oven method of determining moisture prescribed by the Official Association of Agricultural Chemists (2) which requires grinding the sample to pass a 20-mesh sieve prior to oven-drying.

TABLE I

RELATIVE HUMIDITIES OF ATMOSPHERES IN CONTACT WITH SATURATED SOLUTIONS OF VARIOUS SALTS AT 25°C.

Salt	Relative humidity
	%
Lithium chloride	11.1
Potassium acetate	22.5
Magnesium chloride hexahydrate	32.5
Potassium carbonate	43.7
Sodium dichromate dihydrate	53.3
Sodium nitrite	64.4
Sodium chloride	75.4
Potassium chromate	86.4
Ammonium dihydrogen phosphate	92.5

This procedure was not applicable, as the samples would undergo changes in moisture content during grinding, thereby making it impossible to obtain a determination of the moisture content at the time the sample was removed from the conditioning chamber. Moisture contents were therefore determined by placing a sample of approximately 5–10 g. of the rice fraction into a previously dried moisture dish, weighing the sample in the closed moisture dish, drying the sample at 101°–103°C. for 16 hours in a forced draft oven, and reweighing. The covers were replaced on the moisture dishes while they were still in the hot oven, and the covered dishes were cooled to room temperature in a vacuum desiccator containing phosphorus pentoxide as the desiccant. All moisture determinations were made in duplicate, and the results were calculated on the wet basis.

The above-mentioned procedure was used in this investigation because the large number of samples involved at each weighing necessitated the use of the forced-draft oven; slight changes in the time interval of drying had very little effect on the final moisture content; and the slope of the moisture-content curve as a function of time was very nearly horizontal at the time interval chosen.

To compare the results obtained by the above-described method with those obtained by the official method (2), an investigation was made of the effect of state of subdivision, time, and the method of drying on the moisture content of the various rice fractions. For this purpose large samples (100–200 g.) of rough rice, polished rice, and hulls were placed in previously tared crystallizing dishes and the

samples accurately weighed. These samples were then allowed to come into equilibrium with atmospheric conditions of 50% relative humidity at 25°C. for a period of one week after which they were reweighed and ground to pass through a 20-mesh sieve. The grinding was made with a laboratory scale mill situated in the same air-conditioned room in which the samples were stored. Duplicate portions of each of the above-described samples in both the ground and unground state were weighed into previously tared moisture dishes and placed in a forced-draft oven kept at 101°–103°C. This oven was so arranged that the samples could be weighed at oven temperature. Weighings were made at intervals up to 150 hours and the moisture content was calculated from the loss in weight. Bran and polish were also equilibrated at 50% relative humidity and 25°C. and dried as described above. In these cases the state of subdivision was already less than 20-mesh; therefore further grinding was unnecessary.

Other samples of each fraction and its ground portion were weighed into moisture dishes and dried in the vacuum oven at 99°C. Since it was not possible to weigh the samples in the vacuum oven, dry air was admitted to the oven, the moisture dish was sealed, placed in a desiccator over phosphorus pentoxide, cooled, and weighed. The samples were then replaced in the vacuum oven and drying was continued at 99°C. for a total of 145 hours.

Results

Comparison of Vacuum Oven and Air-Oven Moisture Methods. The comparative rates of drying of Rexora variety rice and its fractions determined by the two oven methods of drying are given in Tables II and III. The ground material lost weight in the forced-draft oven much faster than the whole rough rice, polished rice, or hulls, and attained a final moisture content approximately 1% higher than that attained by the unground material. The apparent moisture content of the bran continued to rise throughout the drying period. When dried in a vacuum oven, the ground rice fractions lost moisture more rapidly, but the final equilibrium values attained after 145 hours of drying were in better agreement for both the ground and unground fractions than in the forced-draft oven (Table III).

In the vacuum oven method the values for the apparent moisture content of the bran and polish increased to a maximum value in 12.5 hours, after which there was a slight regain in weight and an apparent decrease in moisture content. The same materials dried in the forced-draft oven continued to lose weight throughout the drying period. These differences in behavior may be attributed to the oil content of these materials. Slight oxidation at the double bonds would cause an

increase in weight and an apparent decrease in moisture content while further oxidation would result in decomposition products which could volatilize and the moisture content would appear to increase.

TABLE II

PERCENTAGE OF MOISTURE REMOVED (WET BASIS) FROM REXORA VARIETY OF RICE AND ITS FRACTIONS DRIED IN A FORCED-DRAFT AIR OVEN AT 101°–103°C. AT VARIOUS TIME INTERVALS

Drying period, hours	Whole rough rice	Ground rough rice	Whole polished rice	Ground polished rice	Whole hulls	Ground hulls	Bran	Polish
⅓	7.4	13.9	7.4	12.5	8.2	13.4	10.2	10.4
⅔	10.0	14.8	10.5	13.4	11.0	14.2	13.4	11.3
1	11.3	15.3	11.7	14.1	12.2	14.5	14.1	11.7
4.5	14.0	16.3	14.2	15.5	13.0	14.8	15.0	12.2
8	14.5	—	14.8	—	—	—	15.2	12.5
11.5	—	16.7	—	16.1	13.2	14.9	—	—
15.5	15.4	—	15.2	—	—	—	15.8	12.7
23.5	—	16.8	—	16.2	13.3	15.0	—	—
28	—	17.0	—	16.4	13.4	15.1	—	—
30.5	15.9	—	15.5	—	—	—	16.4	13.3
48	—	17.1	—	16.4	13.4	15.1	—	—
55	16.2	—	15.6	—	—	—	16.8	13.7
143	16.4	—	15.6	—	—	—	17.6	13.6
168	—	17.3	—	16.5	13.5	15.3	—	—

TABLE III

PERCENTAGE OF MOISTURE REMOVED (WET BASIS) FROM REXORA VARIETY OF RICE AND ITS FRACTIONS DRIED IN VACUUM OVEN AT 99°C. AT VARIOUS TIME INTERVALS

Drying period, hours	Whole rough rice	Ground rough rice	Whole polished rice	Ground polished rice	Whole hulls	Ground hulls	Bran	Polish
1	7.0	12.4	7.7	10.5	12.5	13.1	11.9	8.9
2	10.0	14.0	10.9	12.7	13.2	13.6	13.3	10.6
4	12.4	14.9	13.2	14.1	13.4	14.2	14.0	11.4
7	13.7	15.7	14.4	15.2	14.0	14.5	14.5	11.8
12.5	14.6	16.1	15.1	15.7	14.1	14.7	14.5	11.9
21.5	15.2	16.3	15.5	16.1	14.1	14.8	14.5	11.7
39.5	15.8	16.5	16.0	16.6	14.2	14.9	14.3	11.7
103	16.4	16.5	16.5	16.9	14.2	14.8	14.1	11.8
145	16.5	16.4	16.6	16.8	14.2	14.8	14.1	11.8

The results also indicate that the drying procedure used in this investigation yields numerical values for moisture content in rough and polished rice in good agreement with those obtained by drying ground samples of rough and polished rice in the vacuum oven at 99°C. for 7 hours.

Hygroscopicity of Rough Rice and Mill Products. The effect of length of storage at constant relative humidity at 25°C. on the moisture

content of rough rice and its fractions for artificially dried and naturally dried lots of rice is given in Tables IV–VII and in Figs. 1–4, respectively. The rough rice and head rice consist of whole grains, and the rate of diffusion of moisture to the surface is relatively slow. Probably this is why the rough rice and polished head rice do not attain hygroscopic equilibrium as rapidly as do the bran and hulls. This is an im-

TABLE IV

PERCENTAGE OF MOISTURE (WET BASIS) OF ARTIFICIALLY DRIED ROUGH RICE STORED FOR VARIOUS TIME INTERVALS AT CONSTANT RELATIVE HUMIDITY

Conditioning period, days	Relative humidity of conditioning atmosphere, %								
	11.1	22.5	32.5	43.7	53.3	64.4	75.4	86.4	92.5
1	11.4	11.4	11.6	11.8	11.8	12.1	12.2	12.3	12.6
2	10.2	10.7	10.9	11.5	11.6	12.0	12.5	12.7	13.2
3	9.4	10.0	10.4	11.1	11.4	12.1	12.6	12.9	13.9
4	8.8	9.6	9.9	10.9	11.4	12.1	12.7	13.2	14.4
5	8.2	9.1	9.8	10.7	11.3	12.0	12.8	13.3	14.9
7	7.1	8.3	9.3	10.4	11.1	12.1	12.9	13.6	15.6
9	6.5	7.9	8.9	10.0	11.0	12.1	12.9	13.9	16.1
11	6.2	7.7	8.8	10.0	11.1	12.2	13.0	14.0	16.3
14	5.7	7.4	8.6	9.9	11.0	12.0	13.0	14.4	17.1
17	5.6	7.4	8.6	9.9	11.1	12.1	13.0	14.7	17.5
21	5.2	7.1	8.4	9.7	10.9	12.0	13.1	15.0	17.7
28	4.9	7.0	8.3	9.6	10.8	12.0	13.1	—	18.0
35	4.9	7.0	8.3	9.8	10.9	12.1	13.1	15.3	18.2
42	4.7	—	—	—	—	—	—	15.4	18.2

TABLE V

PERCENTAGE OF MOISTURE (WET BASIS) OF UNDERMILLED RICE FROM ARTIFICIALLY DRIED ROUGH RICE STORED FOR VARIOUS TIME INTERVALS AT CONSTANT RELATIVE HUMIDITY

Conditioning period, days	Relative humidity of conditioning atmosphere, %								
	11.1	22.5	32.5	43.7	53.3	64.4	75.4	86.4	92.5
1	11.9	12.4	12.3	12.7	12.9	13.1	13.3	13.5	13.6
2	10.3	11.5	11.2	12.2	12.7	13.3	13.7	14.0	14.4
3	9.1	10.5	10.6	11.8	12.6	13.3	13.9	14.3	14.8
4	8.1	9.9	10.2	11.5	12.4	13.4	14.1	14.6	15.4
5	7.4	9.2	9.9	11.2	12.4	13.4	14.2	14.7	15.6
7	6.7	8.5	9.4	10.8	12.2	13.4	14.1	15.1	16.3
9	6.2	8.2	9.2	10.7	12.1	13.3	14.2	15.4	16.9
11	6.0	8.1	9.2	10.7	12.2	13.5	14.3	15.6	17.4
14	5.5	7.8	9.0	10.7	12.2	13.4	14.4	15.8	17.9
17	5.3	7.6	8.9	10.7	12.2	13.6	14.5	16.1	18.3
21	5.1	7.5	8.8	10.6	12.2	13.5	14.6	16.4	18.6
28	4.9	7.4	8.9	10.6	—	13.6	14.6	—	19.2
35	4.9	7.4	9.1	10.7	12.2	13.7	14.8	16.8	19.5
42	4.8	—	—	—	—	13.6	14.7	16.8	19.4

TABLE VI

PERCENTAGE OF MOISTURE (WET BASIS) OF BRAN FROM ARTIFICIALLY DRIED ROUGH RICE STORED FOR VARIOUS TIME INTERVALS AT CONSTANT RELATIVE HUMIDITY

Conditioning period, days	Relative humidity of conditioning atmosphere, %								
	11.1	22.5	32.5	43.7	53.3	64.4	75.4	86.4	92.5
1	7.5	8.0	8.4	8.7	9.2	9.3	9.8	10.1	10.5
2	6.8	7.3	7.8	8.5	9.0	9.2	10.2	10.8	11.4
3	6.3	7.0	7.6	8.3	8.9	9.5	10.5	11.2	12.3
4	6.0	6.9	7.5	8.4	8.8	9.5	10.5	11.7	13.0
5	5.9	6.8	7.4	8.0	8.9	9.3	10.6	12.1	13.8
7	5.4	6.6	7.2	8.0	8.8	9.5	10.9	12.6	15.1
9	5.2	6.4	7.0	7.9	8.7	9.6	11.0	13.0	15.6
11	5.0	6.3	7.0	7.9	8.6	9.7	11.4	13.6	17.8
14	4.8	6.2	6.9	7.7	8.6	9.4	11.2	13.8	17.6
17	4.8	6.2	7.0	7.7	8.6	9.6	11.6	15.4	18.2
21	4.7	6.1	6.8	7.7	8.5	9.5	11.6	16.6	18.0
28	4.7	6.1	7.0	7.7	8.6	9.5	11.7	—	17.6
35	4.7	6.1	6.8	7.6	9.1	9.9	12.4	17.0	17.4
42	—	—	—	—	9.0	9.7	—	17.2	17.7

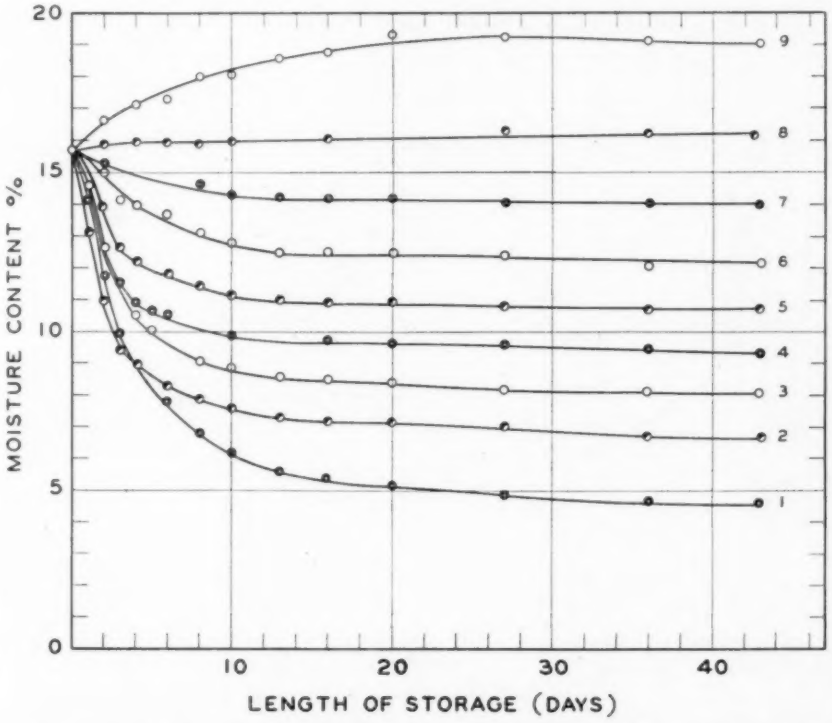

Fig. 1. Effect of length of storage at constant relative humidity on the moisture content of naturally dried rough rice. Relative humidities are: (1) 11.1%; (2) 22.5%; (3) 32.5%; (4) 43.7%; (5) 53.3%; (6) 64.4%; (7) 75.4%; (8) 86.4%; and (9) 92.5%.

TABLE VII

PERCENTAGE OF MOISTURE (WET BASIS) OF HULLS FROM ARTIFICIALLY DRIED ROUGH RICE STORED AT VARIOUS TIME INTERVALS AT CONSTANT RELATIVE HUMIDITY

Conditioning period, days	Relative humidity of conditioning atmosphere, %								
	11.1	22.5	32.5	43.7	53.3	64.4	75.4	86.4	92.5
1	8.4	8.8	9.3	9.8	10.1	10.4	10.5	10.9	10.9
2	7.2	8.0	8.9	9.7	9.8	10.4	10.8	11.2	11.6
3	6.2	7.7	8.5	9.3	9.9	10.4	11.0	11.4	11.9
4	5.9	7.5	8.1	9.0	9.8	10.4	11.0	11.3	12.6
5	5.6	7.0	7.9	8.9	9.7	10.8	11.0	11.6	12.9
7	4.9	6.9	7.6	8.8	9.7	10.5	11.0	12.0	13.5
9	4.5	6.4	7.5	8.7	9.5	10.4	11.0	12.0	13.9
11	4.7	6.4	7.6	8.8	9.7	10.5	11.1	12.2	14.2
14	4.3	6.2	7.4	8.6	9.6	10.4	11.4	12.4	14.6
17	4.2	6.1	7.3	8.4	9.5	10.3	11.1	12.4	14.8
21	4.0	6.1	7.2	8.3	9.6	10.4	11.2	12.6	15.3
28	4.1	5.9	7.2	8.5	9.5	10.4	10.9	—	15.1
35	3.8	5.9	6.9	8.2	9.4	10.4	11.0	12.9	15.1
42	3.8	—	7.0	8.4	—	—	—	12.9	15.2

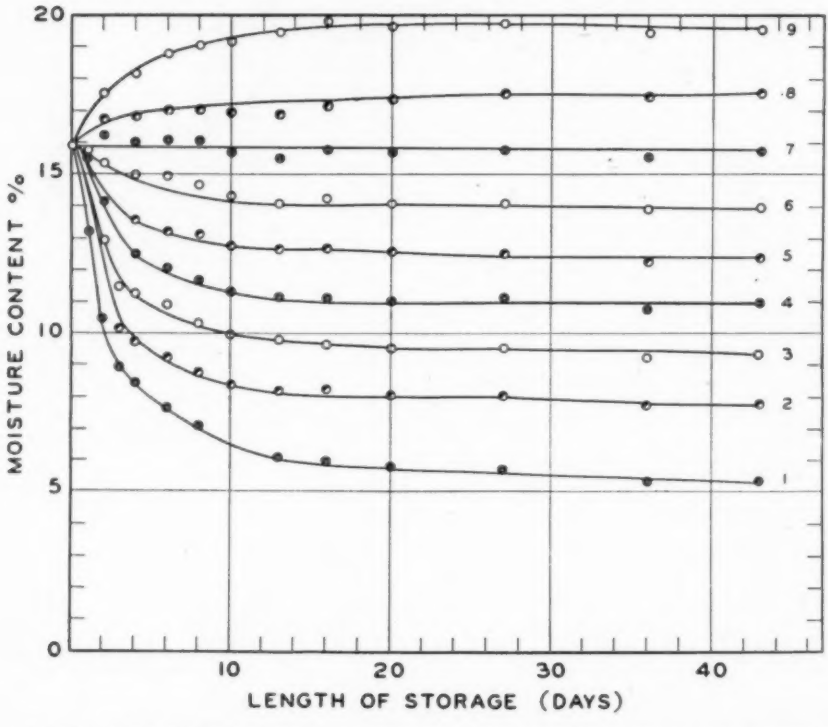

Fig. 2. Effect of length of storage at constant relative humidity on the moisture content of polished rice from naturally dried rough rice. Relative humidities are: (1) 11.1%; (2) 22.5%; (3) 32.5%; (4) 43.7%; (5) 53.3%; (6) 64.4%; (7) 75.4%; (8) 86.4%; and (9) 92.5%.

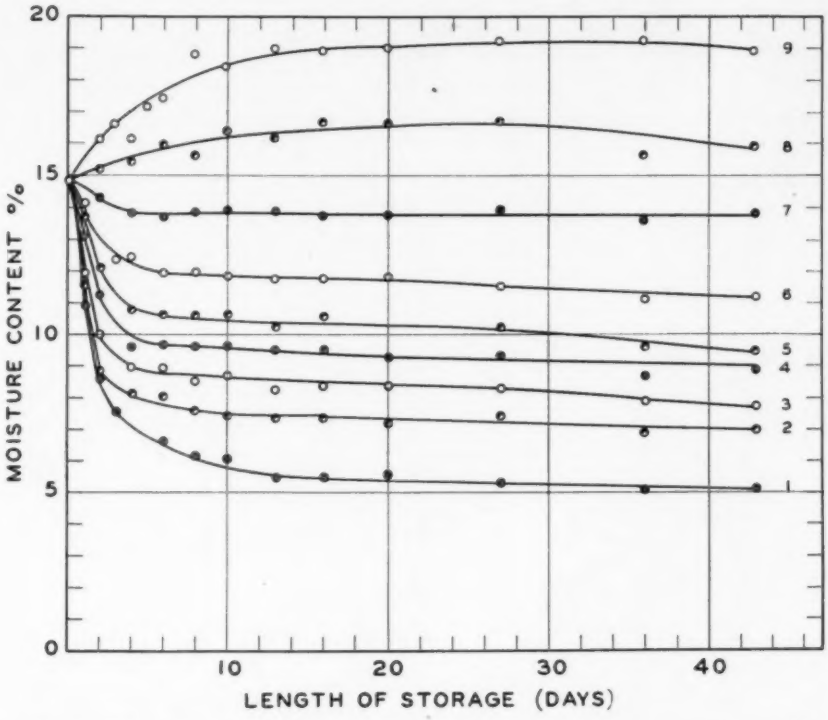

Fig. 3. Effect of length of storage at constant relative humidity on the moisture content of bran from naturally dried rough rice. Relative humidities are: (1) 11.1%; (2) 22.5%; (3) 32.5%; (4) 43.7%; (5) 53.3%; (6) 64.4%; (7) 75.4%; (8) 86.4%; and (9) 92.5%.

TABLE VIII

Percentage of Moisture (Wet Basis) of Various Fractions from Artificially Dried and Naturally Dried Rough Rice in Equilibrium with Atmospheres of Different Relative Humidities

Description of sample	Relative humidity of atmosphere, %								
	10	20	30	40	50	60	70	80	90
Rough rice[1]	4.4	6.5	7.9	9.1	10.4	11.8	13.2	14.8	17.6
Rough rice[2]	4.6	6.6	8.0	9.3	10.6	11.6	12.6	13.8	17.0
Rough rice[3]	—	7.6	9.0	10.2	11.3	12.6	13.8	15.3	18.1
Polished rice[1]	5.2	7.6	9.2	10.5	12.0	13.4	14.8	16.4	18.8
Undermilled[2]	4.6	7.0	8.6	10.0	11.4	12.8	14.2	15.4	18.4
Bran[1]	5.0	6.4	8.0	9.0	10.0	11.0	12.4	14.8	18.0
Bran[2]	4.6	5.8	6.6	7.4	8.3	9.2	10.6	Moldy	—
Hulls[1]	3.7	5.4	6.8	8.1	9.5	10.8	11.8	12.9	15.3
Hulls[2]	3.7	5.4	6.8	7.9	9.1	10.1	10.8	11.6	14.0
Polish[1]	5.3	7.0	8.2	9.2	10.1	11.0	12.4	14.5	18.0

[1] Field-dried rough rice.
[2] Rice which had been artificially dried prior to receipt at mill.
[3] Data of Coleman and Fellows (3) converted to wet basis.

portant consideration in rice drying since, if the outer portion of the rice grain is dried rapidly while the inner part remains relatively wet, strains are set up which produce cracks and checks and subsequently result in many broken kernels during milling. In all cases, however,

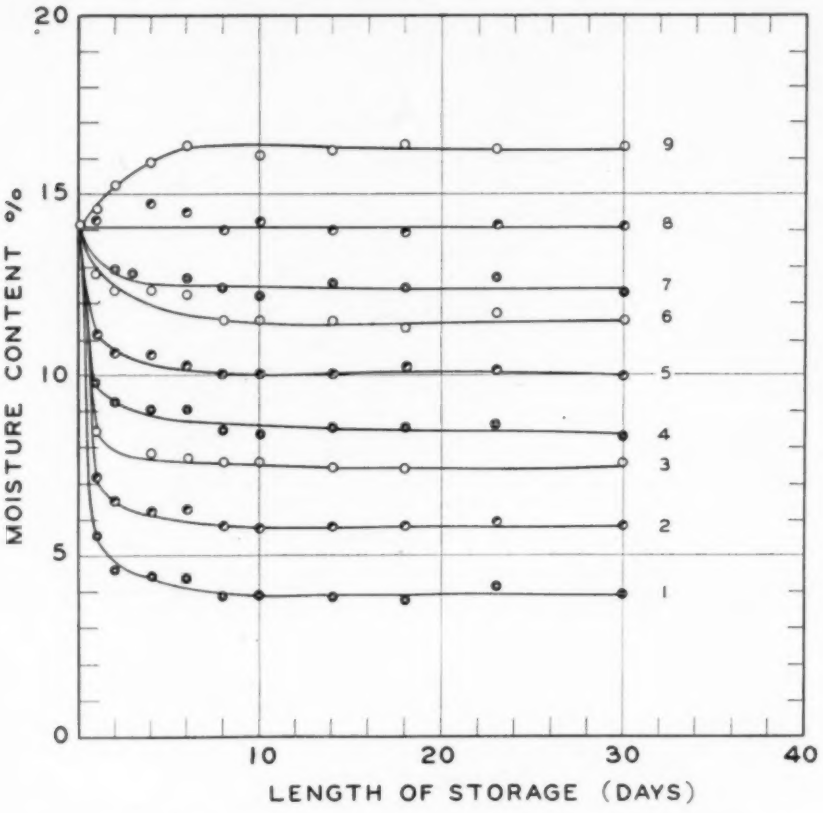

Fig. 4. Effect of length of storage at constant relative humidity on the moisture content of hulls from naturally dried rough rice. Relative humidities are: (1) 11.1%; (2) 22.5%; (3) 32.5%; (4) 43.7%; (5) 53.3%; (6) 64.4%; (7) 75.4%; (8) 86.4%; and (9) 92.5%.

the samples attained hygroscopic equilibrium within a period of three weeks with the exception of those equilibrated over solutions of very high or very low relative humidities.

The equilibrium moisture contents of the samples are given in Table VIII and are shown graphically in Figs. 5 and 6. There were only small differences between the corresponding equilibrium value of naturally dried and artificially dried fractions. In those cases where

the difference in moisture content at a constant relative humidity is significant, the artificially dried sample contains less moisture.

These data can be considered to be in good agreement with those of Coleman and Fellows (3), who reported equilibrium moisture values for three samples of rough rice approximately 1% higher than those of

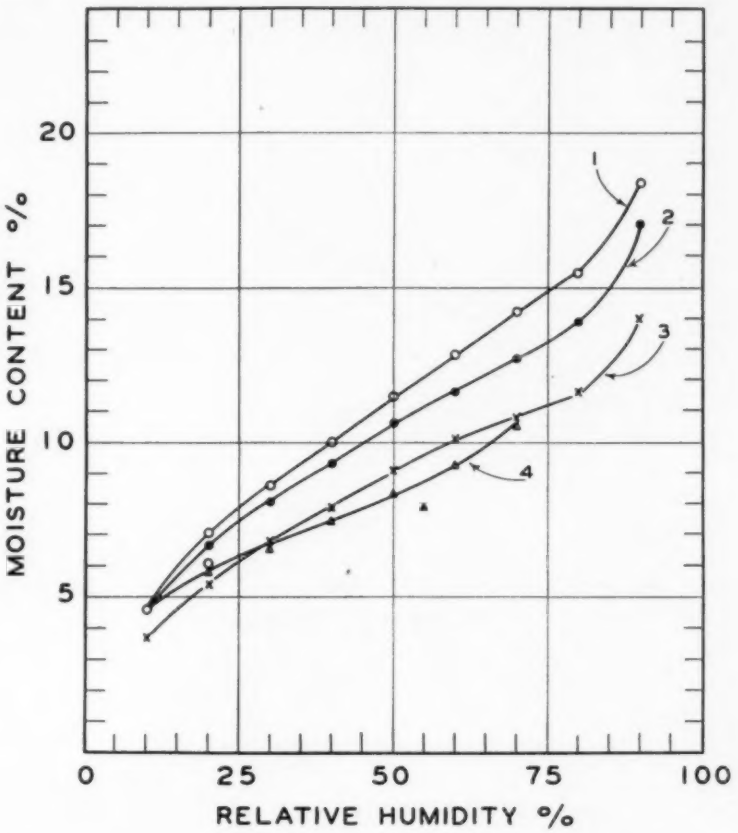

Fig. 5. Moisture content of various fractions of artificially dried rough rice in equilibrium with atmospheres of controlled relative humidity: (1) undermilled; (2) rough rice; (3) hulls; and (4) bran.

the present authors. These higher values are probably explained by the fact that Coleman and Fellows dried their samples at 100°C. in a water oven for 120 hours, whereas the authors used a forced-draft oven for 16 hours. Reference to Table II indicates that in the case of the forced-draft oven method there would be a difference of about 1% in the values obtained with rough rice dried for 16 and 120 hours, respectively.

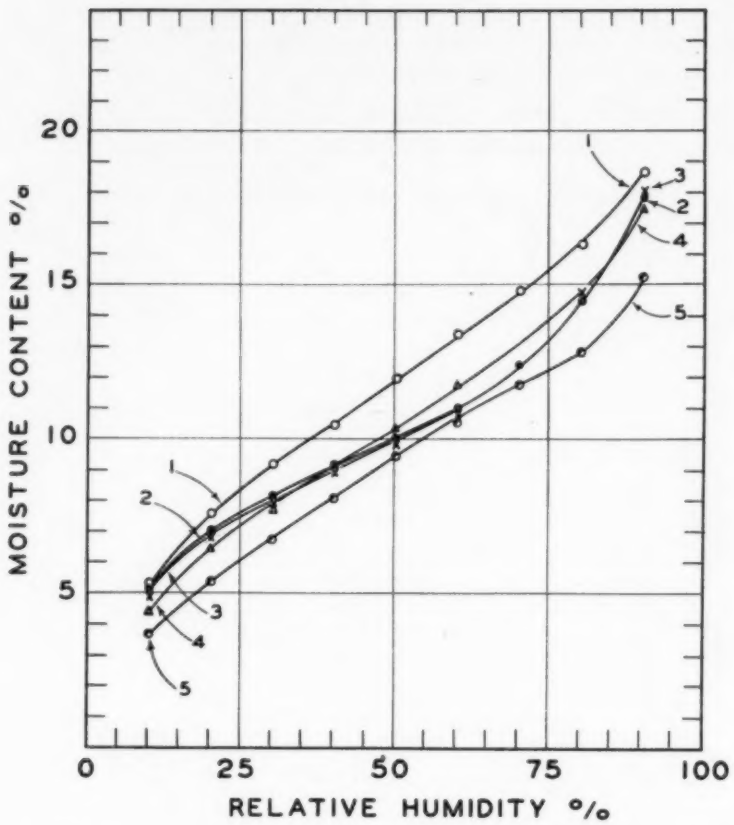

Fig. 6. Moisture content of various fractions of naturally dried rough rice in equilibrium with atmospheres of controlled relative humidity: (1) polished rice; (2) polish; (3) bran; (4) rough rice; and (5) hulls.

Acknowledgment

The rice used in these investigations was furnished through the courtesy of Mr. E. V. Schafer, United Rice Milling Products Company, Inc., New Orleans, Louisiana.

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INFLUENCE OF GROWTH-REGULATING CHEMICALS ON THE MALTING OF BARLEY AND THE COMPOSITION OF MALT [1]

ALLAN D. DICKSON, H. L. SHANDS, and B. A. BURKHART

ABSTRACT

No apparent effect on germination of grain or malt composition was noted when very dilute solutions (0.02–10.0 p.p.m.) of indole acetic acid and naphthalene propionic acid were used in steep or spray water.

In a preliminary malting series using naphthaleneacetic acid and naphthaleneacetamide in concentrations of 2.5–10 p.p.m. as a steeping medium no effect was found in growth or malt composition. In concentrations of 25, 50, 100, and 200 p.p.m. in the steep water, applied to Oderbrucker and Wisconsin Barbless barley, effects were obtained especially at the higher concentrations. The greatest effect was to reduce rootlet development and to increase recovery by approximately 2% with Oderbrucker barley at 200 p.p.m. concentration of naphthaleneacetic acid. Malt extract, diastatic power, alpha-amylase, wort nitrogen/malt nitrogen, and growth were also reduced somewhat by this treatment. The lower concentrations showed less effect on these factors. Wisconsin Barbless was less influenced than Oderbrucker by the treatments. Naphthaleneacetamide treatment resulted in slight increases in malt extract and wort nitrogen/malt nitrogen compared to controls, and in values for rootlet growth and recovery intermediate between the control and the naphthaleneacetic acid treatment.

Three esters of naphthaleneacetic acid that have been used to delay the breaking of dormancy of rose bushes, and that appear to delay hydrolysis of the starch, were applied in vapor form to steeped barley. No marked effect upon the amylase values was apparent although the higher concentration of two esters resulted in increased values for wort nitrogen/malt nitrogen.

During recent years many investigations have been made of the influence of various chemicals on the growth of plants. More studies have been made on phases of plant growth other than germination, and the data are conflicting on the effect of plant-growth-regulating chemicals on this process. Most workers report no effect in very dilute concentrations but an inhibition when higher concentrations are used, or an inhibition at all concentrations (9), (4), (20), (3), (1), (6). Malcher and Zika (12) report that the treatment of barley seed with 0.00625% heteroauxin in most cases gave increased yield of barley and a higher saccharification value of the malt. Crosier *et al.* (5) found that treating seed with dusts containing indolebutyric acid and naphthaleneacetamide increased the percentage of emerged seedlings and total number of heads, especially from shriveled seeds.

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Cooperative between the University of Wisconsin Agricultural Experiment Station and U. S. Department of Agriculture, Bureau of Plant Industry, Soils, and Agricultural Engineering, Division of Cereal Crops and Diseases.

Esters of naphthaleneacetic acid have been used to prevent sprouting of potato tubers, rose bushes, and other plants in storage (6), (14). Hydrolysis of starch is reduced by the treatment, which may indicate inhibition of the amylases. Eyster (8) found that indole-3-propionic, indole-3-butyric, indole-3-acetic, and α-naphthaleneacetic acid reduced diastatic activity *in vitro*. When the diastase was adsorbed on activated charcoal, addition of growth-substances increased diastatic activity over the control.

This paper represents studies carried out over several years to determine the effect of growth-regulating chemicals upon germination during malting and on the composition of the malt. Some of these compounds have also been applied to barley plants in the field at various stages of growth. The effects upon growth, yields per acre, and malting quality will be presented in a later paper.

Materials and Methods

Two varieties of barley, Oderbrucker and Wisconsin Barbless, grown on the University Farm at Madison, Wisconsin, were used. The first series of experiments were carried out on barleys from the 1938 crop, and the later work was done on barleys grown in 1943.

In most of the experiments reported in this paper, the growth-regulating chemicals were applied in the steep water. All barleys were steeped to 45% moisture at 16°C. and malted for 6 days at this temperature and moisture content. The malts were kilned a total of 32 hours according to the schedule: 8 hours, 25°; 4 hours, 35°; 12 hours, 45°; 4 hours, 55°; and 4 hours, 65°C. The rootlet loss and percentage recovery values were obtained by careful weighing before and after cleaning, and all calculations were made on the dry weight basis. The methods of the American Society of Brewing Chemists (2) were used for growth and malt analysis. Growth index as used here represents the sum of values obtained by multiplying the number of kernels in a classification by the higher limit, e.g., no. in 0–¼ × ¼ + no. in ¼–½ × ½ + no. in ½–¾ × ¾ + no. in ¾–1 × 1. Alpha-amylase was determined by the Olson, Evans, and Dickson (16) 20°C. modification of the Sandstedt, Kneen, and Blish method (19), and the values calculated as 20° dextrinizing units (17).

Experimental

Indoleacetic Acid and Naphthalenepropionic Acid in Dilute Solutions. Indoleacetic acid at 10.0 and 0.1 p.p.m. and naphthalenepropionic acid at 2.0 and 0.2 p.p.m. concentration were used for steeping grain of the two barley varieties. One set of samples was steeped 12 hours in solutions of the growth-regulating chemicals and 24 hours

in water. Another set of samples was steeped 36 hours in the growth-regulating solutions. During the malting period the samples were maintained at 45% moisture by spraying with water twice a day.

The summary data for this series are presented in Table I, as averages for the two varieties. It is apparent that there are no significant differences in growth index, recovery, or the three malt composition factors that can be attributed to the use of the chemicals. Additional samples of the two barleys were steeped as described above, and in addition indole-3-acetic acid at 100 p.p.m. and naphthalenepropionic acid at 20 p.p.m. were used instead of water as a daily spray to maintain the individual samples at the proper moisture level. Those samples receiving the additional amounts of the two chemicals in higher concentrations showed little difference in growth or malt composition.

TABLE I

INFLUENCE OF INDOLEACETIC ACID AND NAPHTHALENEPROPIONIC ACID AS VERY DILUTE SOLUTIONS IN THE STEEP ON GROWTH AND COMPOSITION OF THE MALTS—AVERAGES OF ODERBRUCKER AND WISCONSIN BARBLESS BARLEYS

Material used	Concentration	Hours steeped in hormone solution	Extract dry basis %	Diastatic power °L.	Wort N. / Malt N. %	Growth index	Recovery of malt from barley %
Indole 3 acetic acid	0.1 p.p.m.	12	73.6	152	35.3	81.4	90.2
Indole 3 acetic acid	0.1 p.p.m.	36	73.5	152	34.4	78.6	90.3
Indole 3 acetic acid	10.0 p.p.m.	12	73.6	148	34.8	80.1	90.1
Indole 3 acetic acid	10.0 p.p.m.	36	73.7	146	35.3	80.6	90.3
Water steep		0	73.4	150	34.6	81.8	89.8
Naphthalenepropionic acid	0.02 p.p.m.	12	73.8	154	35.0	81.4	89.9
Naphthalenepropionic acid	0.02 p.p.m.	36	73.6	158	34.4	82.4	91.1
Naphthalenepropionic acid	2.0 p.p.m.	12	73.6	154	34.8	80.1	90.1
Naphthalenepropionic acid	2.0 p.p.m.	36	73.0	145	34.4	80.8	90.3

Naphthaleneacetic Acid and Naphthaleneacetamide in Several Concentrations. Single maltings were made of Wisconsin Barbless samples from the 1943 crop that had been steeped for 36 hours in solutions containing 2.5, 5.0, and 10.0 p.p.m. of naphthaleneacetic acid, and 5.0 and 10.0 p.p.m. of naphthaleneacetamide. A control steeped in water was malted at the same time. The maximum variations for malt factors between the six malt samples were as follows: extract 0.6%, wort nitrogen/malt nitrogen 2.6%, diastatic power 10°L., alpha-amylase 1.0 20° unit, growth index 7.3, rootlet loss 0.5%, and recovery 0.5%. These values are within the limits of variation between replicate maltings and indicate no effect of the growth substances at these concentrations.

In view of the results obtained above, a more comprehensive experiment was planned using duplicate maltings of two varieties and four concentrations of each chemical, 25.0, 50.0, 100.0, and 200.0 p.p.m. Preliminary tests with the two highest concentrations indicated that germination was not completely inhibited, but that rootlet

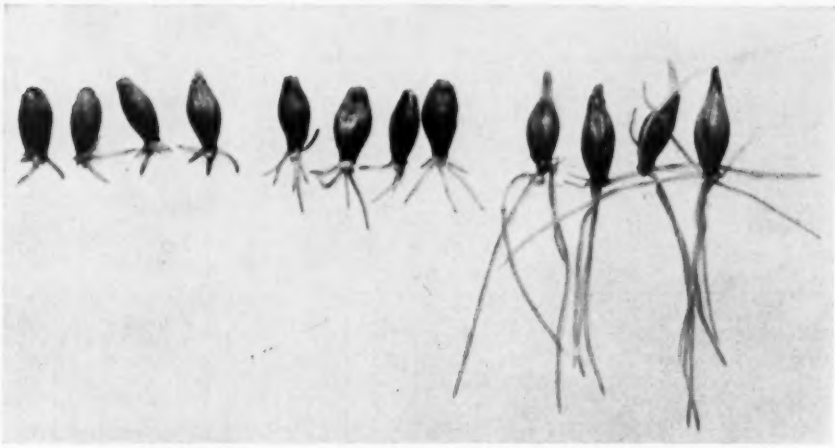

Fig. 1. Effect of naphthaleneacetic acid (200 p.p.m.) and naphthaleneacetamide (200 p.p.m.) on the blotter germination of Oderbrucker barley in comparison with control (right).

development was not normal. Fig. 1 shows a typical effect of 200 p.p.m. of naphthaleneacetic acid and naphthaleneacetamide on rootlet development during germination on blotting paper. Duplicate Oderbrucker samples were steeped 34 hours, and Wisconsin Barbless samples 36 hours in solutions of the two chemicals at the four concentrations. Control samples were steeped in water. The solutions were drained off, and the samples malted as described earlier. The moisture during germination was maintained by spraying the samples with tap water. Fig. 2 shows representative kernels from the treatment with 200 p.p.m. of the chemicals and the control after 4 days germination.

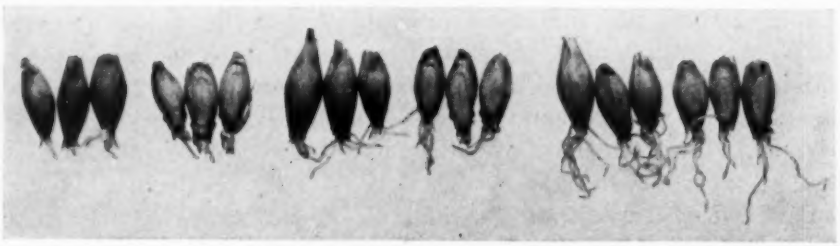

Fig. 2. Effect of naphthaleneacetic acid (200 p.p.m.) and naphthaleneacetamide (200 p.p.m.) on the malting of Oderbrucker barley compared with control water steep after 4 days germination.

The data, as averages of duplicate maltings, for recovery of malt from barley, rootlet loss, extract, wort nitrogen/malt nitrogen, diastatic power, and alpha-amylase are presented as histograms for the individual factors in Figs. 3 to 5.

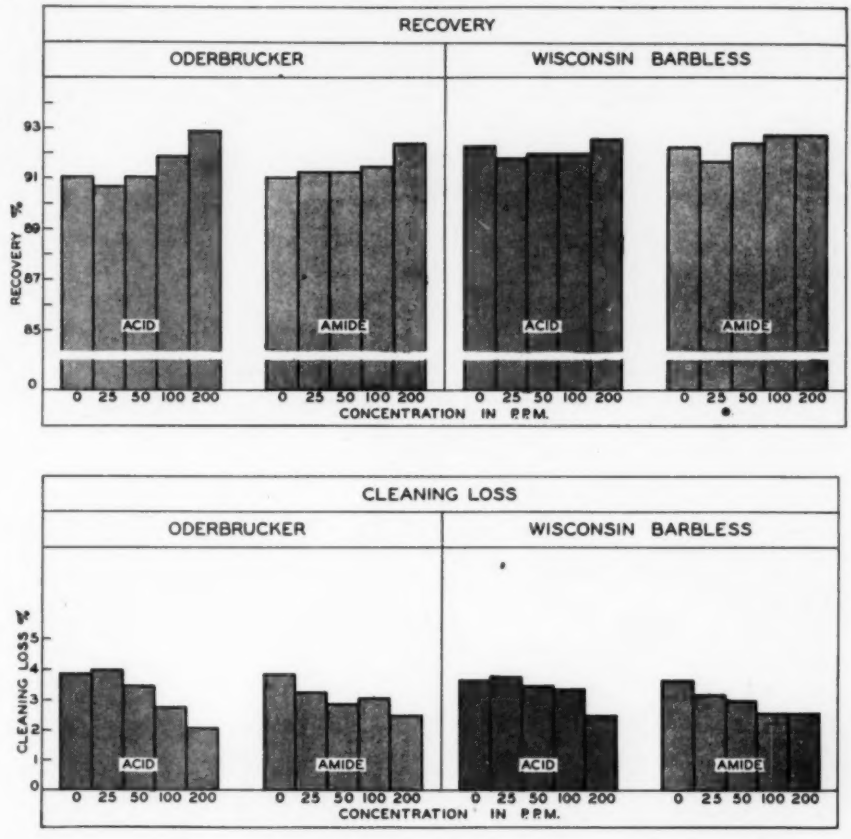

Fig. 3. Effect of naphthaleneacetic acid and naphthaleneacetamide on recovery of malt and cleaning loss.

The detailed data for growth index are not presented, since differences from water steeps are small in relation to variability between duplicate maltings, and are of doubtful significance. The acid treatment resulted in an average growth index of 78, in comparison to 83 for the controls. The amide stimulated growth slightly, producing an average index of 93, which resulted in a significantly different effect of the two growth-regulating chemicals. This effect was similar for the two varieties.

Recovery of malt from barley was increased by the two higher concentrations of both chemicals as indicated in Fig. 3. This increase

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was greater with naphthaleneacetic acid than with the amide, and was considerably greater for Oderbrucker than for Wisconsin Barbless. With Oderbrucker, 200 p.p.m. of the acid resulted in an increased recovery of 1.8% over the control; the same concentration of the amide showed an increase of 1.3%. With Wisconsin Barbless, the greatest

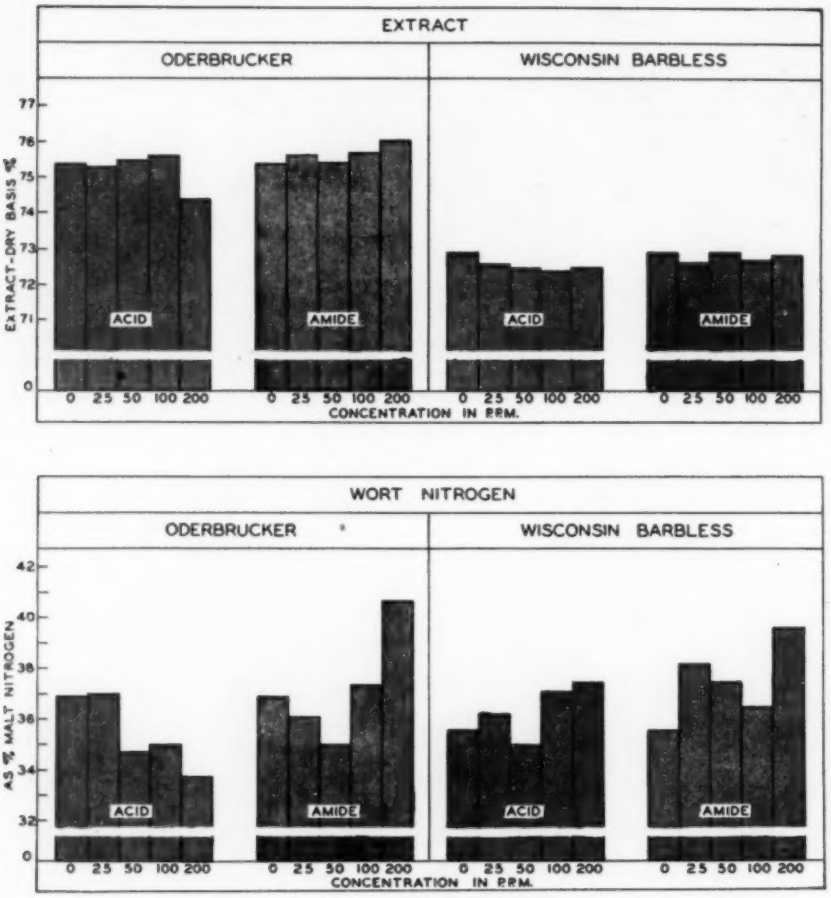

Fig. 4. Effect of naphthaleneacetic acid and naphthaleneacetamide on malt extract and wort nitrogen as per cent of malt nitrogen.

increases over the control were less than 1.0% and were somewhat greater for the amide than the acid.

The recovery of malt from barley is influenced by losses in steep, respiration, and in cleaning. The last consists primarily of rootlets. With Oderbrucker, the reductions in malt cleaning loss by treatment with the two chemicals were as great as the increases in recovery mentioned above. Respiration of Oderbrucker was uninfluenced by

treatment, and recovery increased because of lower rootlet loss. Wisconsin Barbless reacted somewhat differently to treatment. In this variety, respiration was increased slightly by treatment. However, cleaning loss was sufficiently reduced by treatment to result in greater recovery than the control.

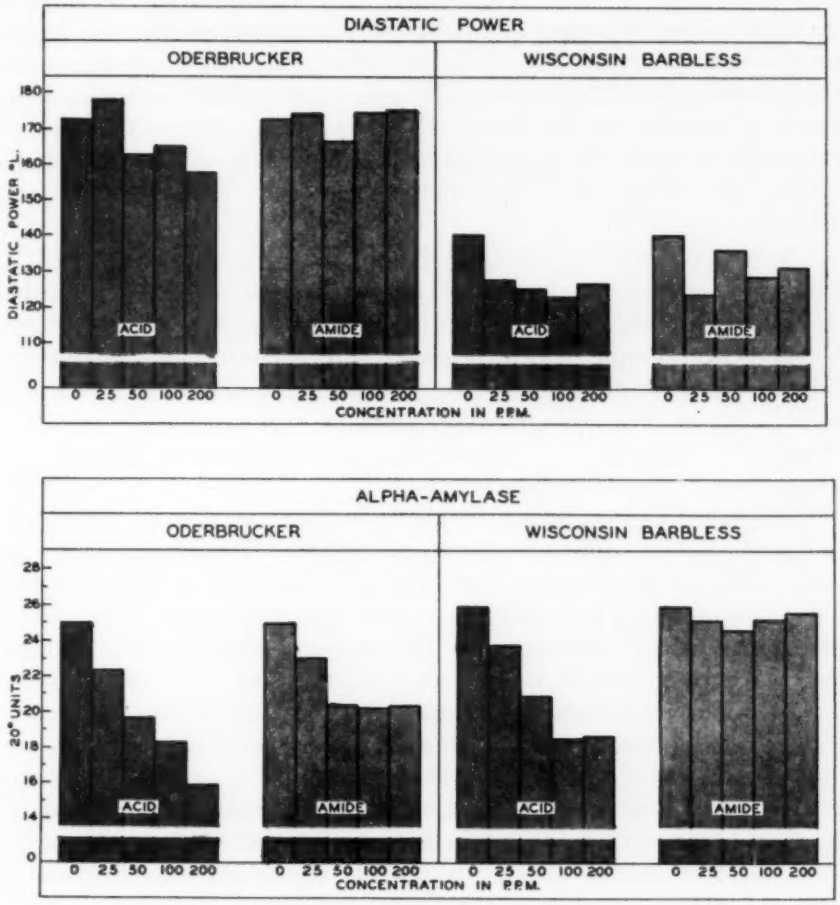

Fig. 5. Effect of naphthaleneacetic acid and naphthaleneacetamide on malt diastatic power and alpha-amylase.

Four quality factors are presented in Figs. 4 and 5 in the discussion of effects of growth-regulating chemicals on malt composition. The two chemicals used produced different effects. Treatment with the acid showed no effect on extract of Oderbrucker malts, but resulted in a small decrease in the Wisconsin Barbless malts. The amide, especially at the 200 p.p.m. concentration, produced increased extract in Oderbrucker malts, but showed no effect on the other variety. Although

significant in relation to the variability between duplicate maltings, the differences in all cases were relatively small, 0.5 to 0.7%.

The wort nitrogen/malt nitrogen ratio has been considered to be a measure of modification in malt, and also a rough measure of proteolytic activity. For this factor also, the chemicals and varieties reacted differently. With Oderbrucker the acid resulted in a small decrease in wort nitrogen at the higher concentrations, but the amide produced an increase, especially at 200 p.p.m. Both chemicals increased the wort nitrogen/malt nitrogen values of Wisconsin Barbless malts, especially at the higher concentrations. The maximum increases over the control samples were obtained at 200 p.p.m. of the amide and were 3.8 for Oderbrucker and 4.0 for Wisconsin Barbless.

Most treatments with growth-regulating chemicals resulted in small but significant reductions in diastatic power values of the malts, as indicated in Fig. 5. An exception was the amide treatment on the Oderbrucker sample which produced no effect. The greatest decreases below the control samples were 15°L. for Oderbrucker samples and 16°L. for Wisconsin Barbless samples, representing decreases of 9 and 11% respectively. These were produced by the highest concentrations of naphthaleneacetic acid.

The effect of treatment on alpha-amylase activity of malts was similar to that on diastatic power, except that the treatment with the amide showed no reduction with Wisconsin Barbless malts. The acid treatment resulted in greater decreases than the amide in the Oderbrucker samples, especially at the higher concentrations. Alpha-amylase activity was depressed more than diastatic power by the treatments. The maximum reductions under the control for Oderbrucker and Wisconsin Barbless were 36 and 28% respectively and were obtained with 200 p.p.m. of the acid.

Volatile Esters Applied as Vapors. Several esters of naphthaleneacetic acid when applied in vapor form have been found to prevent the breaking of dormancy of potatoes and certain shrubs when in common storage. These treatments delay the hydrolysis of starch and the production of reducing sugars. Since this could be an effect on the amylase systems of the plants, it seemed desirable to study, in a preliminary way, the influence of these compounds on malting and malt composition.

Methyl and ethyl esters of naphthaleneacetic acid in concentrations of 0.5 and 2.0 g. per 1,000 cubic feet and 2-methyl naphthylacetic acid at the rate of 2.0 g. per 1,000 cubic feet were vaporized and applied to duplicate Oderbrucker barley samples which had been steeped previously to 45% moisture. The calculated quantities of the esters

were dissolved in 5 ml. of absolute alcohol and this poured onto the bottoms of 2-gallon friction top pails. The steeped barleys were placed in the pails, the lids placed on tightly, and the chemicals volatilized by placing the pail on a hot plate for 5 minutes. The pails were then placed in a 16° chamber and the steeped barleys allowed to stand in contact with the vapors for 12 hours. At the end of this period the samples were removed from the pails and malted for 5½ days at 16°C. and 45% moisture. The control samples were given the same treatment except that 5 ml. of absolute alcohol only were used. The data for growth, recovery, and certain of the malt factors as means of duplicate maltings are presented in Table II. The effects of all

TABLE II

INFLUENCE OF VOLATILIZED ESTERS OF NAPHTHALENEACETIC ACID APPLIED TO STEEPED ODERBRUCKER BARLEY ON THE GERMINATION AND COMPOSITION OF THE MALT

Materials used	Concentration g. per 1000 cu. ft.	Growth index	Recovery of malt from barley %	Loss in cleaning malt %	Extract dry basis %	Wort N. / Malt N. %	Diastatic power °L.	Alpha-amylase 20° dext. units
Ethyl ester naphthaleneacetic acid	0.5	79.7	91.8	3.5	75.9	36.5	170	24.9
Ethyl ester naphthaleneacetic acid	2.0	84.4	91.1	3.8	76.3	37.5	178	25.0
Methyl ester naphthaleneacetic acid	0.5	83.9	91.9	3.2	75.1	37.5	171	24.8
Methyl ester naphthaleneacetic acid	2.0	78.2	92.0	3.3	75.3	41.8	168	23.2
2-Methyl-1-naphthylacetic acid	2.0	76.9	91.9	3.3	75.5	36.1	167	23.6
Control	0.0	80.9	91.9	3.4	75.5	35.7	167	24.2

treatments were small and only a few were significant. The higher concentration of the ethyl ester produced the highest growth index and rootlet loss, with the concomitant low recovery; the highest value for extract, diastatic power, and alpha-amylase, and a relatively high value for wort nitrogen/malt nitrogen. All treatments resulted in increased values for wort nitrogen, but only the higher concentration of the methyl ester gave an appreciable increase. Further studies with the esters on more samples would be required to verify these results.

Discussion

Although the literature dealing with the effect of growth-regulating chemicals on germination and growth of cereals is somewhat conflicting, most studies have shown no stimulation of germination at very low concentrations, but inhibition or retardation at higher concentrations. Marmer (13), Weimer (20), and others present evidence that germinat-

ing seeds themselves contain relatively large quantities of auxins or heteroauxins. The results obtained on malting of barley with several different chemicals are in agreement with these findings. The retardation of barley germination and growth appeared to be confined primarily to rootlet development, with only slight effects on elongation of the plumule or acrospire. This is in agreement with the reaction of wheat seedlings to 100–200 p.p.m. of indole-3-acetic acid as found by Loo and Tang (11) and others. Among the explanations given for the effect of these compounds is an increase in the respiration of wheat seedlings associated with an inhibition of growth by 50 p.p.m. of indole-3-acetic acid (18). Hsueh and Lou (10), on the other hand, found that high concentrations (0.1%) of 2–4 D inhibited aerobic respiration and germination of barley. The rough measures of respiration used in this study indicated either no effect or a slight stimulation of respiration of barley during malting by naphthaleneacetic acid and naphthaleneacetamide.

Eyster (8) and Miller and Kneen (15) report an inhibition of amylase action by growth-regulating chemicals *in vitro*. The decreases in amylase activity obtained in this study are probably due to physiological effects upon the germination process rather than the effect of small residual quantities of the chemicals remaining on the malt.

Preliminary experiments with those esters of naphthaleneacetic acid that delay the breaking of dormancy of tubers and shrubs in storage indicated no effect on the germination or amylase activity of barley malts. Denny, Guthrie, and Thornton (6) reported an inhibition of the hydrolysis of starch and production of reducing substances by these materials when they were applied to potato tubers. This effect, possibly upon the amylases, may be an indirect one of controlling physiological processes leading to the hydrolysis.

The effects of the higher concentrations of naphthaleneacetic acid and naphthaleneacetamide upon germination, while primarily of interest in relation to physiology of germination, indicate certain practical applications in malting. By the choice of chemical and concentration, it should be possible to increase recovery of malt from barley by 1 to 2% without excessive deleterious effects upon the malt characteristics. There is some evidence that one might improve malt quality over a water-steeped sample, and still obtain an improved recovery of approximately 1%. Plant scale tests of the use of growth-regulating chemicals would be required to determine the practicability of their commercial use, if not undesirable for other reasons. These data at least indicate the desirability of investigating other chemicals and concentrations applied to more barley varieties.

Acknowledgments

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SIGNIFICANCE OF LOAD-EXTENSION TESTS IN ASSESSING THE BAKING QUALITY OF WHEAT FLOUR DOUGHS [1]

P. HALTON [2]

ABSTRACT

An apparatus is described with which the rate of extrusion of dough through an orifice under constant pressure can be measured. With it the absorptions of different flours can be assessed by determining the water contents necessary to give doughs having the same rate of extrusion.

An extensometer is described with which load-extension curves can be made on dough.

Doughs made either with or without yeast soften with age and it is necessary to allow for this in comparative baking or load-extension tests. This softening, which is shown to be equivalent to an increase in the water content of the dough, can be compensated for by adjusting the water contents of doughs made from different flours so that they all have the same extrusion time at the period when they are baked or tested on the extensometer.

As a result of this softening the force required to stretch an unyeasted dough decreases with increasing age of the dough. With a yeasted dough the reverse occurs and the stretching force increases. It is suggested that this is not due to a tightening of the dough during fermentation but to modifications in its chemical and physical condition which result in increased work hardening of the dough when stretched. The fermenting dough decreases in extensibility and at the same time the relaxation time increases. It is this change in the relaxation time which is the basis of dough ripening and improved baking quality. It is shown, in this connection, that the fermentation necessary for a given decrease in extensibility and the fermentation time necessary for optimum quality bread bear similar relationships to the yeast content of the dough.

Experimental evidence is presented to show that bread quality is a function of the tensile strength and of the relaxation time of the dough. The tensile strength of the dough is shown to be the basis of flour strength and determines the breadmaking potentialities of the flour, i.e., the quality of bread that can be obtained under optimum conditions. The relaxation time of the dough, on the other hand, is a measure of the condition of the dough and thus largely determines the quality of bread the dough will actually produce. The tensile strength of dough is a function of the wheat from which the flour was milled and does not appear to be affected by normal fermentation, improver treatment, or mechanical manipulation of the dough. On the other hand, the relaxation time is considerably modified by the various chemical and physical processes which form the basis of breadmaking.

Both the tensile stress and the relaxation time of dough can be assessed from a load-extension test.

[1] Manuscript received June 7, 1948.

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A wheat flour dough has certain characteristics normally associated with solids, but in other respects it resembles a liquid. When, for example, a piece of dough is stretched and then released it shows the elastic recovery of a solid. If stretched to any appreciable extent, however, this recovery is not complete and a permanent change in shape results. This nonrecoverable stretch is due to the dough having flowed like a liquid. This fluid nature of dough is also seen when a piece is left to rest on a board and flows out under its own weight.

Halton and Scott Blair (6) studied this dual nature of dough by stretching, under constant force, cylindrical test pieces floated on a bath of mercury. When the stretching force had been applied for a given time the increase in length of the dough cylinder was measured. The applied force was then removed to allow elastic contraction to take place. By this means the total stretch was divided into its two components, the elastic or recoverable stretch and the viscous or nonrecoverable stretch. Such tests formed the basis for determining the rigidity modulus and the viscosity of dough.

From experiments made with flours of varying baking quality it was found that the viscosity and modulus values depended on a number of factors including the water content and age of the dough. In addition the following two generalizations were arrived at: 1. When the correct absorptions, found for each flour from baking trials, were used, doughs made with various flours differed considerably in their viscosities but had similar values for modulus. This indicated that measurements of rigidity modulus might be used for assessing the water absorption of different flours. 2. When data obtained on a number of flours were examined it was found that the better the breadmaking quality of the flour the higher was the ratio of the viscosity to the rigidity modulus of the dough. The authors explained how this ratio, which is equivalent to Maxwell's "relaxation time," (9) determined the "spring" of "elasticity" of the dough, a factor which bakehouse experience had shown to be closely related to baking quality.

Halton and Scott Blair also investigated the connection between the physical properties of dough and its "ductility" or "extensibility," another factor which bakehouse experience had shown to be related to baking quality.

When the viscosity of dough is determined by a stretching test the actual value obtained depends on the stress applied to the dough and also on the strain to which the dough is subjected. When the stress is increased the viscosity falls, a phenomenon known as "structural viscosity." On the other hand, when the strain is increased the viscosity rises, the dough exhibiting the characteristic of "work hardening" shown by some metals. If the applied force remains constant

during stretching, the stress and strain may both increase, the stress, i.e., force per unit cross section, increasing due to the thinning of the test sample. When both stress and strain increase, the viscosity of the dough is modified by both work-hardening and structural viscosity.

Halton and Scott Blair obtained indications that variations in structural viscosity were associated with variations in the extensibilities of different doughs. Experiments showed, however, that this only applied to some flours and that measurements of structural viscosity, as then made, did not give a true indication of dough extensibility in all cases. It was realized that one possible reason for this was that, with the technique used, the stresses and strains were much smaller than those operating in a fermenting dough under conditions which lead to tearing and rupture. The present paper deals with the continuation of this work in which the modified technique of Schofield and Scott Blair (11), more suitable for the study of dough properties at large stresses and strains, has been used.

The viscosity and rigidity modulus of dough can be calculated from the equations:

$$\text{viscosity} = \frac{\text{stress}}{\text{viscous strain in unit time}},$$

$$\text{rigidity modulus} = \frac{\text{stress}}{\text{elastic strain}}.$$

In these equations three factors are involved, i.e., stress, strain, and time. In the technique used by Halton and Scott Blair, stress and time of application of the stress were kept constant while variations in strain were measured. In the modified technique used in the experiments now to be described, strain and time were kept constant while variations in stress were measured.

Materials and Methods

The apparatus used (see Fig. 1) consisted of a mercury bath on which a cylinder of dough, approximately 0.7 cm. in diameter, was floated. One end of the dough was fastened to a piece of cork connected by a cotton thread to a winch operated by a constant speed

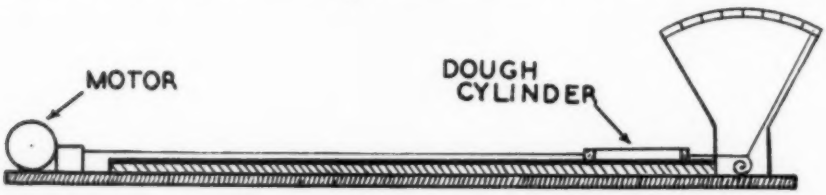

Fig. 1. Mercury bath extensometer—diagrammatic.

motor. The other end of the dough was fastened to a second piece of cork connected by a cotton thread to a load meter. This meter contained a coiled spring to which was attached a pointer operating over a graduated scale. When the motor was started, the dough cylinder stretched, the increase in length being proportional to the time of stretching less a small correction due to the shift in position of that end of the dough attached to the load meter. The force built up during stretching was read off at intervals on the scale of the load meter, one unit on the scale being approximately equivalent to 1 g. From the data so obtained load-extension curves such as is shown in Fig. 2

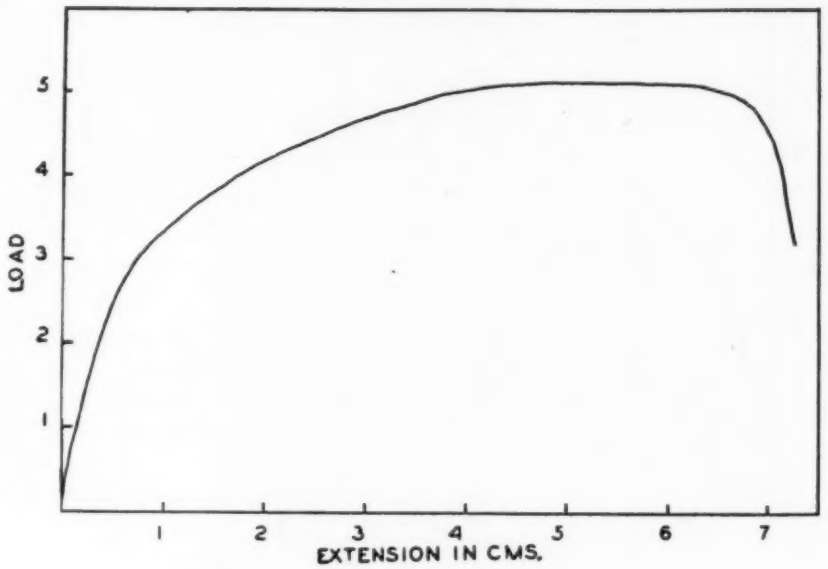

Fig. 2. Load-extension diagram obtained on mercury bath extensometer.

could be obtained. Such curves were, however, of little value since their dimensions depended upon the water content of the dough and no differentiation between the viscous and elastic deformations was possible. In practice, therefore, the dough was stretched for a given time, the direction of the motor was then reversed to allow the dough to relax, and when this was complete and the load meter reading had returned to zero, the motor was once more put in its forward position and the stretching of the dough continued until rupture occurred. By this procedure data were obtained from which curves, such as is shown in Fig. 3, were constructed.

This curve shows the change in load and extension during the three stages of the test, A–B being the first extension, B–C the relaxation,

and C–D the final stretch ending at the point D when the dough rapidly tore and then broke.

At the time corresponding to point B in the curve the dough cylinder had stretched 1.25 cm., this being partly elastic and partly viscous. On relaxation an elastic recovery of 0.75 cm. took place leaving a permanent elongation of 0.5 cm., this last being a measure of the viscous flow. By this separation of the dough extension into its two components the viscosity and modulus of the dough could be calculated. The actual values in this particular case were viscosity $= 0.51 \times 10^6$ and modulus $= 0.88 \times 10^4$ C.G.S. units.

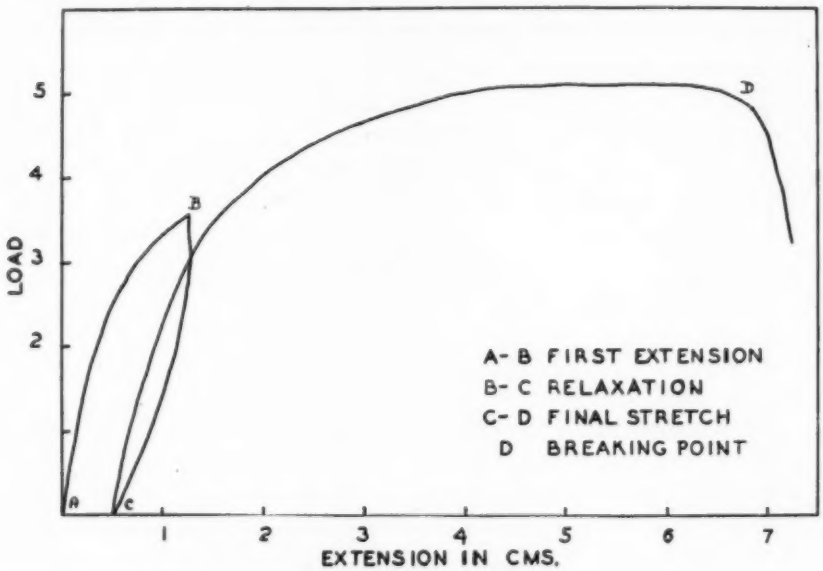

Fig. 3. Load-extension diagram with relaxation of dough.

It will be realized that these values applied to the dough during the stretching from A–B. By relaxing the dough after different periods of stretching, however, the change in the physical properties throughout the stretching period could be assessed and thus the over-all effect of work hardening and structural viscosity determined.

When load-extension curves had been obtained on a number of flours a different use for these curves became apparent. It has been pointed out earlier in this paper that when the physical properties of doughs made from different flours are to be compared, the water contents should be so adjusted that the doughs all have a similar modulus. This adjustment was made for the load-extension tests and an examination of the resultant curves showed that in general the better the baking quality of the flour the greater was the height of the curve.

In other words the load developed during stretching was an indication of the breadmaking quality of the dough.

The reason for this is that the force required to stretch a dough depends on both its viscosity and its modulus. If a number of doughs having similar values for modulus are tested, any differences in their viscosities will therefore be reflected in corresponding differences in the force required to stretch them. These variations will therefore

Fig. 4. Self-recording extensometer.

run parallel with variations in the viscosity-rigidity modulus ratio or relaxation time of the dough and, from the work of Halton and Scott Blair, will therefore correlate with variations in baking quality.

The significance of this finding lay in the possibility that a single load-extension test might give a measure of two important dough properties: the load giving an assessment of the elasticity of the dough and the extension an assessment of the ease with which the dough tore when stretched.

The examination of load-extension curves for a number of flours showed that while variations in the heights of the curves agreed with what was to be expected from baking tests on the flours, the length of the curves did not correlate well with the extensibilities of the doughs as assessed in the bakehouse. Indications were obtained that this might be due to the method of preparing the dough cylinders by extrusion. Trials were therefore made with balls of yeasted dough which could be

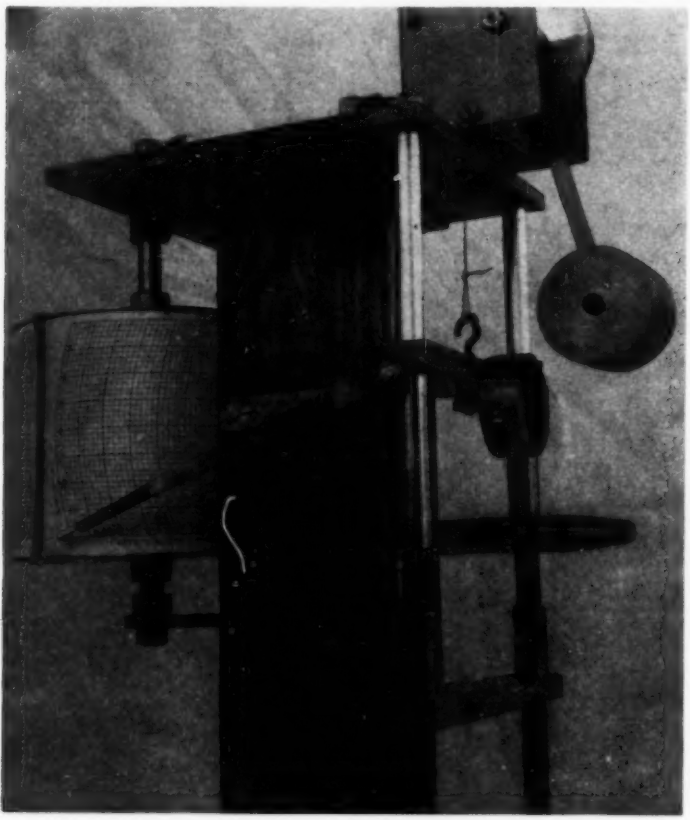

Fig. 5. Self-recording extensometer.

handled much the same as the larger bakehouse doughs. Experience soon showed that a much truer measure of extensibility was being obtained and in view of this the simple extensometer (5) shown in Figs. 4 and 5 was constructed to enable load-extension tests to be made on this shape of test sample.

The ball of dough is impinged on two semicircular pegs which lie in contact. These pegs are each attached to a crosshead which is positioned by and can slide up and down two vertical guides. The

crosshead of the top peg is attached to a balance while the crosshead of the bottom peg is attached to a threaded sleeve which is moved up or down by a threaded rod activated by an electric motor. This rod also operates through gearing to turn a recording drum. The upper crosshead engages one end of a pivoted arm, on the other end of which is a pen which presses against the recording drum.

When the dough has been impinged on the two pegs the motor is switched on. The bottom peg then moves downwards at constant rate and the recording drum revolves. As the dough is stretched the force built up pulls the top peg downwards against the opposing balance weight, the degree of movement being proportional to the force. This movement is recorded and magnified by the upward movement of the pen writing on the chart wrapped round the recording drum. When the dough has been extended to the point when it breaks, the force pulling on the top dough peg is released and as this peg moves upwards, the recording pen completes the load-extension curve drawn on the chart.

This machine, like the Chopin Alveograph (1) and the Brabender Extensograph (10), is only suitable for comparative tests on different doughs. Actual measurements of dough viscosity and elasticity cannot be determined from the curves. Because of this some method other than the direct measure of the rigidity modulus had to be used for assessing the water contents at which doughs made from different flours should be compared.

TABLE I

VISCOSITY AND RIGIDITY MODULUS OF DOUGHS HAVING SAME EXTRUSION TIME

Flour	Viscosity	Modulus
No.	*C.G.S. units*	*C.G.S. units*
1	0.7×10^6	1.0×10^4
2	0.5×10^6	0.9×10^4
3	0.4×10^6	1.0×10^4
4	0.4×10^6	1.0×10^4
5	0.3×10^6	1.0×10^4
6	0.2×10^6	1.0×10^4

The method adopted was to fix these water contents so that all doughs had the same rate of extrusion through an orifice, a method previously used by Jago (7) (and later by Stamberg and Bailey, 12) for determining the water absorptions of different flours. In the work of Halton and Scott Blair (6) in which dough cylinders were prepared by extrusion from a dough gun it was thought that the rate of extrusion was determined by the viscosity of the dough. Later experiments, however, showed that the rate of extrusion was dependent more

on the modulus than on the viscosity. This is illustrated by the data given in Table I. These figures were obtained on doughs made from different flours, the water content in each case being adjusted so that all the doughs had the same rate of extrusion. It will be seen that although the viscosities of the doughs varied considerably there was little variation in their rigidity moduli.

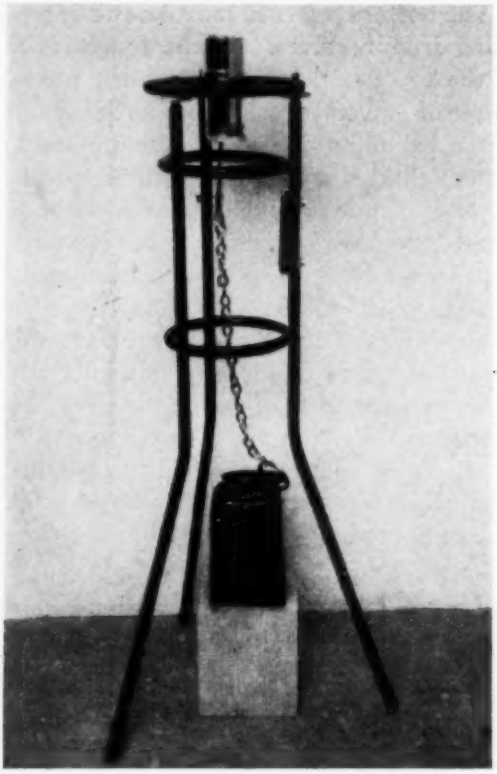

Fig. 6. Water absorption meter.

From these findings it followed that a constant rate of extrusion could be used for assessing the water content at which a dough should be examined on the extensometer.

The dough gun constructed for determining the water absorptions of flours is shown in Figs. 6 and 7. It consists of a hollow cylinder closed at one end by a plate in the center of which is a hole through which the dough is extruded. The cylinder passes through the center of a circular plate resting on leveling screws threaded on three supporting uprights. The dough is forced out of the cylinder by a piston attached to a rod which passes through the dough and through the

orifice. The bottom of this rod is connected by a pin to a collar to which is also attached a chain carrying a weight. The pin forms a pointer which moves over the scale attached to one of the supporting uprights.

The dough to be tested is put into the cylinder of the dough gun. The piston is placed on top and the weight attached to the piston rod.

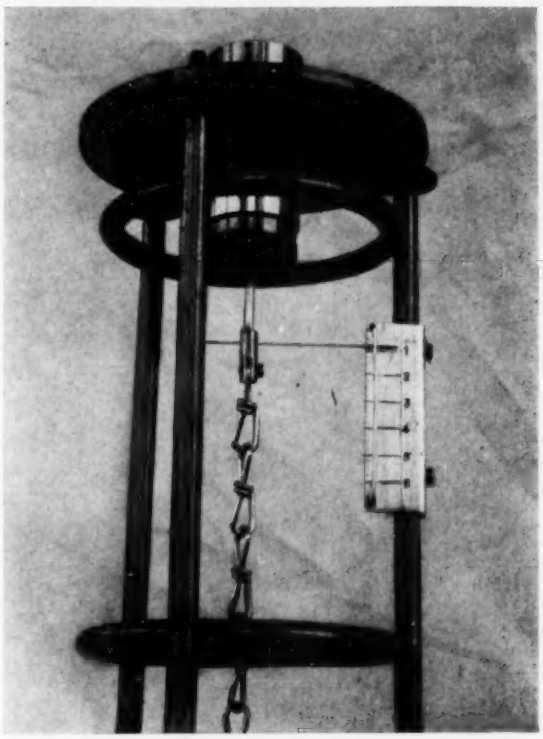

Fig. 7. Water absorption meter.

The dough is extruded through the annular space formed by the piston rod passing through the orifice in the bottom plate of the cylinder. The time for the pointer to move over the scale is measured.

The logarithm of the extrusion time is lineally related to the water content of the dough. Based on this finding a special slide rule was constructed, the time scale of which is logarithmic in form. When the extrusion time for a dough of known water content has been measured, the absorption corresponding to the standard extrusion time is calculated by the use of the slide rule.

The water absorption, corresponding to a constant rate of extrusion, is used in making the doughs to be stretched on the extensometer.

For these tests a parent dough is made from 280 g. flour. This is scaled off into 75-g. pieces which are left to rest under glass jars. Twenty-five minutes before they are tested the doughs are passed once through the molder shown in Fig. 8. This machine is a modified

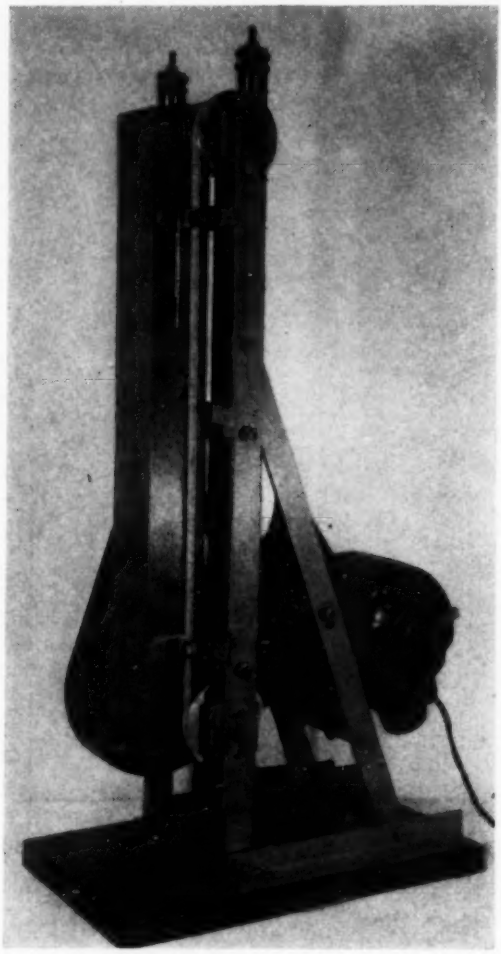

Fig. 8. Dough molder.

version of the punching machine described by Malloch and Hopkins (8). It consists of a framework supporting rollers over which runs an endless vertical belt driven by an electric motor. On one side of the belt is a pressure plate which keeps the belt in contact with a wooden tunnel. This is of semicircular cross section decreasing in radius from the top to the bottom.

At the end of the 25-minute relaxation period after molding the test doughs are stretched on the extensometer. While experiments have been made on yeasted and nonyeasted doughs, the data obtained on the former are generally of more value for interpreting the importance of the physical properties of dough in determining baking quality. Some of the factors involved and their effect on the extensometer curves and on baking quality will now be considered.

Discussion

Water Absorption. A correct measure of the water absorption of a flour is essential if a true assessment of its quality is to be obtained by baking or physical dough tests. When determining this absorption, however, allowance must be made for the fact that during the period between making and baking or testing, the dough may soften.

Halton (4) showed that during this period the fall in the viscosity and rigidity modulus of the dough and, what is of greater significance, the change in the ratio of their values were the same as those resulting from an increase in the water content of the dough. Thus after resting the dough had, in effect, a higher free water content than when freshly made, a change similar to that resulting from syneresis. This view of the softening of dough with age is supported by the work of Baker, Parker, and Mize (2) on the liquors obtained by subjecting doughs to the action of a Sharples supercentrifuge. The amount of liquor they obtained increased when the dough rested after making. They also found that this increase was greater for yeasted than non-yeasted doughs which is in keeping with the author's finding that the softening as measured by the fall in the extrusion time is greater when the dough contains yeast.

The physical condition of the dough and therefore the quality of the loaf it will give is modified by this softening. It is therefore necessary to allow for it, which can be done by determining the extrusion time of the dough, not when it is freshly made but at a time corresponding to that at which the dough would be put into the oven or tested by physical means. The procedure used by the author is to make two or three small doughs from the same flour but with different water contents and to leave these to stand for the necessary time before measuring their extrusion times. The doughs are made with or without yeast, depending on the information required. The points relating the water contents of the doughs when made to the logarithms of their extrusion times lie on a straight line from which the water content necessary to give a dough having the standard rate of extrusion can be read off. By this means the water contents of all doughs, whether made for physical or baking tests, are adjusted so

that when stretched on the extensometer or baked they have the same extrusion time.

The degree of softening of a dough with age, as shown by the fall in its extrusion time, depends on a number of factors including the variety and quality of the wheat from which the flour was milled as well as the percentage extraction of the flour and its content of damaged starch. A dough made from a low extraction flour milled from a strong, sound grist may soften by the equivalent of only 0.5% in absorption during 3 hours' fermentation. During the same time a dough made from a flour of 85% extraction milled from a medium strength grist may soften by the equivalent of 7% in absorption.

Fermentation. Data from extensometer curves obtained on unyeasted doughs of different ages but made from the same flour are given in Table II. It will be seen that as the time between making and

TABLE II

EFFECT OF AGE OF UNYEASTED DOUGHS ON EXTENSOMETER CURVES AND EXTRUSION TIME

Age	Height	Length	Product	Extrusion time
Hour	*cm.*	*cm.*		*Secs.*
1	2.9	24	70	92
2	2.6	25	64	84
3	2.3	27	62	76

testing the dough increased, the height of the curve decreased, its length increased, and the product figure of height × length decreased.[3] These changes in the shape of the curve (like the fall in the extrusion time) suggest a softening of the dough similar to that which results from increasing its water content.

If the change in dough with age, as reflected by the change in the extensometer curve, were entirely due to an increase in its free water content, it should be possible to compensate for it by a suitable reduction in the water added to the dough when made. To test this point a number of doughs were made from the same flour but with varying water contents. The extrusion times of the doughs were measured when they were 1, 3, and 5 hours old and from the data so obtained the water contents necessary to give doughs having the same extrusion time when 1, 3, or 5 hours old were determined. Thus when freshly made the 5-hour dough had the lowest water content and the highest

[3] This applies to doughs made from flours treated with nitrogen trichloride or ammonium persulfate as well as untreated flours. With bromated flours, however, the height of the curve may rise despite the fact that the extrusion time of the dough decreased. This difference in the behavior of bromated doughs is undoubtedly due to the fact that the improving action of potassium bromate is progressive with time. As a consequence, the relaxation time of the dough will tend to increase progressively due to the increasing effect of the bromate and this may more than offset the effect due to the progressive softening of the dough. This time factor in the bromate effect is shown up by the work of Munz and Brabender (10) on the relaxation of dough after molding.

extrusion time while the 1-hour dough had the highest water content and the lowest extrusion time. After standing for their appropriate times each dough had the same extrusion time and, in addition, when stretched on the extensometer they gave similar curves. These tests indicate, therefore, that the changes in the extensometer curves, obtained on a nonyeasted dough as it ages, reflects a softening due to an increase in the free water content of the dough. The degree of softening and the increase in free water can be determined from the change in the extrusion time of the dough.

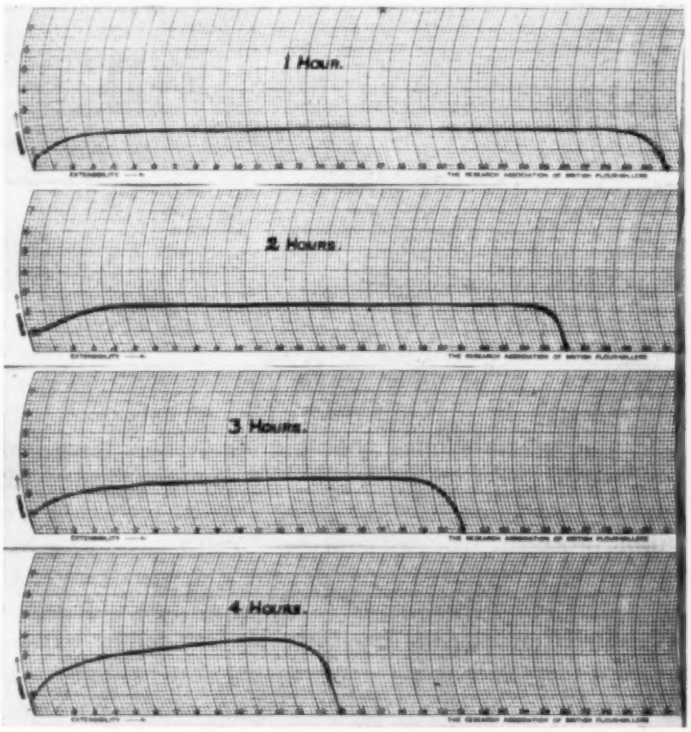

Fig. 9. Effect of fermentation of dough on extensometer curve.

Extensometer curves on yeasted doughs fermented for 1, 2, 3, and 4 hours are shown in Fig. 9. It will be seen that with increasing age of the dough the height of the curve increased and its length decreased, the opposite to what occurred with nonyeasted doughs. This might be taken to indicate that the yeasted doughs became tighter and not softer with age. On the other hand, the data for these doughs given in Table III show that the product figure (height × length of curve) and the extrusion time both decreased with increasing age, suggesting that the dough softened.

When interpreting the results of physical tests on dough it is necessary to bear in mind that the technique of the test may modify the physical characteristics of the dough. This is certainly true of stretching tests which normally work harden and thus toughen the dough. As a consequence the shape of the extensometer curve depends both on the condition of the dough before it is stretched and also on its response to stretching. The extrusion times for both yeasted and nonyeasted doughs fall as the doughs age, suggesting that both soften.

TABLE III

EFFECT OF AGE OF YEASTED DOUGHS ON EXTENSOMETER CURVES AND EXTRUSION TIME

Age	Height	Length	Product	Extrusion time
Hour	*cm.*	*cm.*		*Secs.*
1	2.0	30	60	90
2	2.3	26	59	78
3	2.7	21	57	72
4	3.7	15	55	69

Bakehouse experience also suggests that doughs when left undisturbed, i.e., not molded or stretched, soften. On the other hand, when molded they toughen, so their final characteristics depend on the balance of the two processes. The difference in the yeasted and nonyeasted dough, as reflected by the difference in their extensometer curves, can be explained by assuming that the nonyeasted dough toughens when stretched to the same or decreasing extent with increasing age, whereas the yeasted dough work hardens to an increasing extent as fermentation proceeds.

If a series of, say, four yeasted doughs have their water contents adjusted so that when they are 1, 2, 3, and 4 hours old, respectively, they have similar extrusion times, the corresponding extensometer curves are not identical, as with nonyeasted doughs, but show a progressive increase in height and decrease in length with increasing fermentation. On the other hand, the product figure does remain constant, indicating that the fall in the value of this figure with increasing age reflects a softening of the dough. As with nonyeasted doughs, the degree of softening can be assessed, from the change in extrusion time, in terms of an increase in water content.

While the difference in the aging characteristics of yeasted and unyeasted doughs may be due, at least in part, to the chemical changes that accompany fermentation and which modify the oxidation-reduction systems in the dough, they can also be explained in terms of the physical history of the dough. The unyeasted dough remains static during the rest period between making and stretching. The

yeasted dough, on the other hand, is subjected to stresses resulting from the gas generated during fermentation and which stretches it. The stretching proceeds at a slow rate compared with the stretching on the extensometer or that due to molding and this allows time for the relaxation due to viscous flow to keep pace with the stretching. In consequence the dough does not work harden as when stretched at a much more rapid rate. Despite this, important changes may occur in the dough. Links in the protein network may be broken, the weaker links giving first so that as fermentation proceeds only the

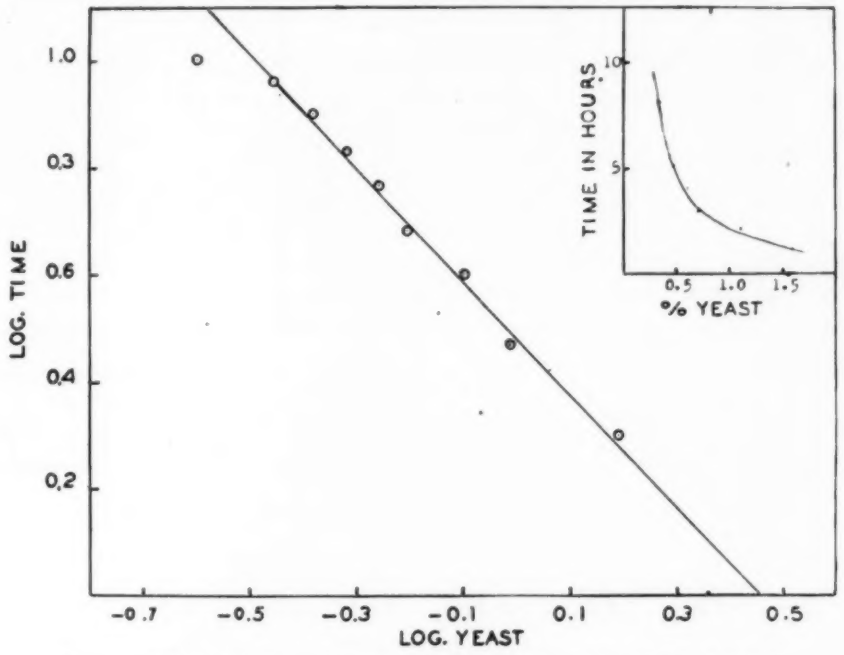

Fig. 10. Relation between yeast content of dough and fermentation time for best loaf.

stronger links remain attached. Thus as fermentation continues the dough work hardens to an increasing extent when it is stretched, this being reflected in extensometer curves of increasing height. As more and more of the links are broken the dough becomes more brittle and less extensible; thus the increase in the height of the curve is accompanied by a decrease in its length.

In bakehouse parlance a dough is said to ripen as fermentation proceeds, this resulting in improved bread quality. This ripening is undoubtedly the result of the changes in dough characteristics resulting from the chemical and physical processes accompanying fermentation and which are reflected in a progressive fall in the extensibility of the dough.

Fisher and Halton (3) in studying the changes in bread quality resulting from fermentation showed that the relationship between the yeast content of a dough and the fermentation time required to give optimum quality bread was logarithmic. Their curves illustrating this relationship are given in Fig. 10. A similar relationship holds between the yeast content of a dough and the time required for the extensibility to fall to any given value. Curves illustrating this are given in Fig. 11. These are based on data obtained from extensom-

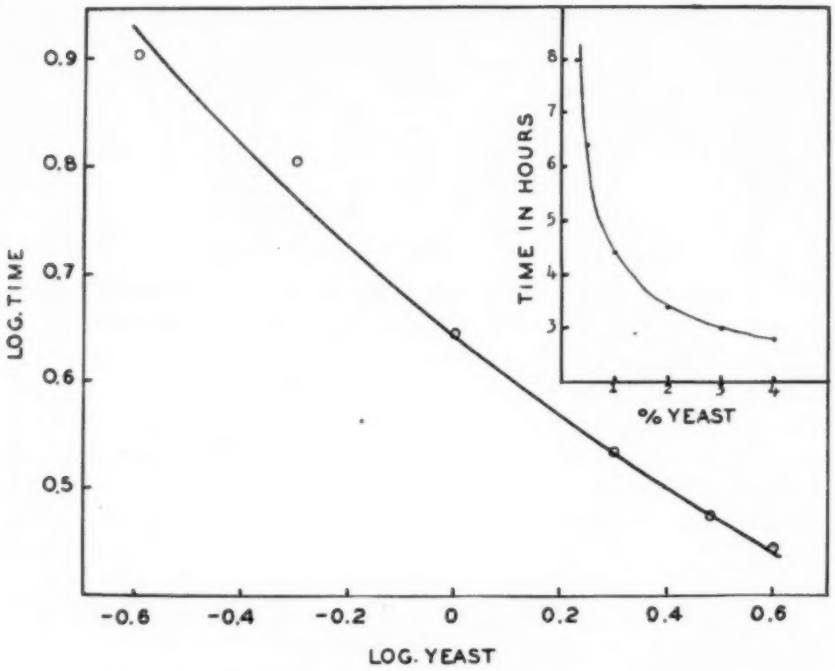

Fig. 11. Relation between yeast content of dough and fermentation time for constant extensibility.

eter curves made on doughs fermented to different lengths of time. For each yeast content the fermentation time at which the extensibility reached a value of 16 was used in constructing the curves.

The fact that the lengths of fermentation necessary to give optimum quality bread, on the one hand, and a given value of dough extensibility, on the other, are in similar relationship to the yeast content of the dough suggests that the ripening of the dough is related to the decrease in extensibility. As explained above, this decrease in extensibility is accompanied by an increase in the work hardening of the dough when stretched. This increased work hardening raises the

TABLE IV

WORK HARDENING IN TERMS OF CHANGE IN VISCOSITY AND RIGIDITY MODULUS

	Flour 1	Flour 2	Flour 3
	C.G.S. units	*C.G.S. units*	*C.G.S. units*
Viscosity after 1st stretch	1.6×10^6	1.3×10^6	0.8×10^6
Viscosity after 2nd stretch	2.6×10^6	2.5×10^6	4.8×10^6
Viscosity after 3rd stretch	3.8×10^6	4.4×10^6	11.0×10^6
Modulus after 1st stretch	0.7×10^4	0.8×10^4	1.2×10^4
Modulus after 2nd stretch	0.7×10^4	0.8×10^4	1.2×10^4
Modulus after 3rd stretch	0.7×10^4	1.0×10^4	1.9×10^4

viscosity and also the relaxation time of the dough.[4] In other words, the correctly fermented dough responds well to the stretching action of molding and, in addition, softens more slowly after molding, i.e., has a longer relaxation time. The improvement in bread quality, up to an optimum, which parallels the increased fermentation of a dough is thus a reflection of the increase in the relaxation time of the dough.

Improvers. The effects of three chemical improvers, potassium bromate, nitrogen trichloride, and ammonium persulfate, on fermented doughs is shown by the data obtained from their extensometer curves

TABLE V

EFFECT OF ADDITION OF IMPROVERS TO FLOUR ON EXTENSOMETER CURVES

	Height	Length	Product
	cm.	*cm.*	
Flour A + 0 p.p.m. potassium bromate	2.0	19	38
Flour A + 10 p.p.m. potassium bromate	2.4	16	38
Flour A + 20 p.p.m. potassium bromate	2.8	14	39
Flour A + 30 p.p.m. potassium bromate	3.2	12	38
Flour B + 0 g. nitrogen trichloride per 280 lb. flour	2.5	16	40
Flour B + 3 g. nitrogen trichloride per 280 lb. flour	2.7	14.5	39
Flour B + 6 g. nitrogen trichloride per 280 lb. flour	2.9	13.5	39
Flour B + 9 g. nitrogen trichloride per 280 lb. flour	3.1	12.5	39
Flour C + 0 oz. ammonium persulfate per 280 lb. flour	2.0	22	44
Flour C + ½ oz. ammonium persulfate per 280 lb. flour	2.4	17.5	42
Flour C + 1 oz. ammonium persulfate per 280 lb. flour	3.3	13	43

and given in Table V. The general effect is the same as that which results from fermentation; the dough becomes more elastic and less extensible, the product figure remaining unaltered. Within limits,

[4] This is illustrated by the data given in Table IV. These data were obtained by stretching cylinders of dough on the mercury bath apparatus. Each test piece was stretched and then relaxed, the viscosity and rigidity modulus being calculated from the changes in length of the sample. This was repeated twice. The data show that the work hardening of the dough was due to an increase in its viscosity, any change in the modulus being small by comparison. Since the viscosity increased to a much greater extent than the modulus, the relaxation time also increased.

fermentation and improvers treatment are complementary, a dough having a short fermentation needing a high treatment and one having a long fermentation, a low treatment. (An unyeasted dough requires a very heavy treatment to reduce its extensibility to the value of a fermented dough.)

When a flour has been overtreated either with chemicals or by heat, or has been overfermented, the product figure falls and this is accompanied by a deterioration in bread quality.

Molding. When a dough has been correctly fermented and treated with an improver, it is brought to the condition when it shows optimum response to the beneficial effects of molding. The importance of molding is illustrated by the difference in the loaves shown in Fig. 12.

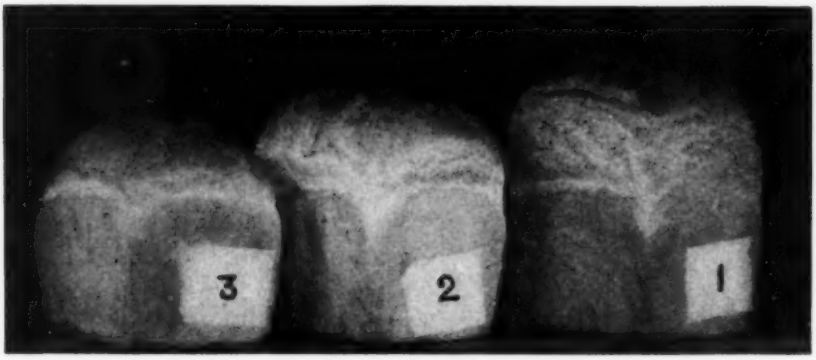

Fig. 12. Effect of molding on loaf quality.

These were made from the same flour and the doughs had identical water and yeast contents, were given the same length of fermentation, but differed in the severity with which they were molded, flour No. 1 being well molded, No. 2 more lightly handled, and No. 3 handled as little as possible.

The beneficial action of molding depends on the stretching of the dough which, by work hardening, increases its elasticity, therefore giving a more rigid structure. The efficiency of the molding process, whether given by hand or by machine, is thus an important factor in determining either bread quality or the shape of the load-extension curves. This must be considered when correlating the results of baking tests with physical tests made on the dough.

Flour Strength. Correct fermentation, improver treatment, and mechanical manipulation all condition the dough, enabling optimum quality bread to be made from any one flour. These processes bring out the potential capabilities of the flour, but they do not alter its fundamental strength.

To appreciate the meaning of flour strength in terms of the physical properties of the dough, further consideration must be given to the

experiments described earlier in this paper in which load-extension curves were obtained for cylinders of dough stretched on the mercury bath instrument.

These load-extension curves were translated into stress-strain curves using the following calculations.

If when the dough cylinder of original length l and radius of cross section r had been stretched to length l_1, the load meter reading was Δ, then:

1. the load in dynes was $981\Delta a$ where a = force in grams necessary to cause unit deflection on the meter;
2. the cross section of the dough cylinder was $\pi r^2 \frac{l}{l_1}$;
3. the shearing stress per unit cross section was therefore:

$$\frac{981\Delta a}{3\pi r^2 \frac{l}{l_1}} = \frac{981\Delta a \times l_1}{3 \times \pi r^2 l} = \frac{\text{Load} \times \text{length}}{3 \times \text{volume}} .$$

4. the strain was $\int_l^{l_1} \frac{dl}{l} = 2 \cdot 3 \log_{10} \frac{l_1}{l}$.

By plotting the calculated values of stress against strain, curves such as is shown in Fig. 13 were obtained. It will be seen that the stress on the dough increased in value until the dough rapidly tore and then broke. From the calculations given above the value of the maximum stress is given by the value of $\frac{\text{load} \times \text{length}}{3 \times \text{volume}}$ at the time when the dough ruptured. Thus for similar-sized test pieces of dough, variations on the maximum stress figures run parallel with variations in the load $\times$ length product figures.

Experience with the extensometer has shown that the stronger the flour the greater is this product figure. It would follow, therefore, that the maximum value of the stress built up during the stretching of the dough is a measure of the strength of the flour. In other words, the strength of a flour is determined by the tensile strength of the dough.

Earlier in this paper, data, obtained from extensometer curves, were given for doughs containing increasing amounts of improvers. Attention was given to the fact that the product figure was not changed by improver addition. The significance of this will now be apparent. The fact that the improver did not change the product suggests that these chemicals have no effect on the fundamental strength of the flour. This applies to quantities of improver that are beneficial to baking quality. Overtreatment lowers the tensile strength of the dough and therefore the product figure. The results of tests described earlier in this paper showed that the product figure, for both unyeasted and

yeasted doughs, decreased in value as the doughs aged. Evidence was presented to show that this was due to a virtual increase in the water content of the dough, a change which resulted in its becoming softer and having a lower extrusion time. When the water contents of doughs, made from the same flour, were adjusted so that each, when

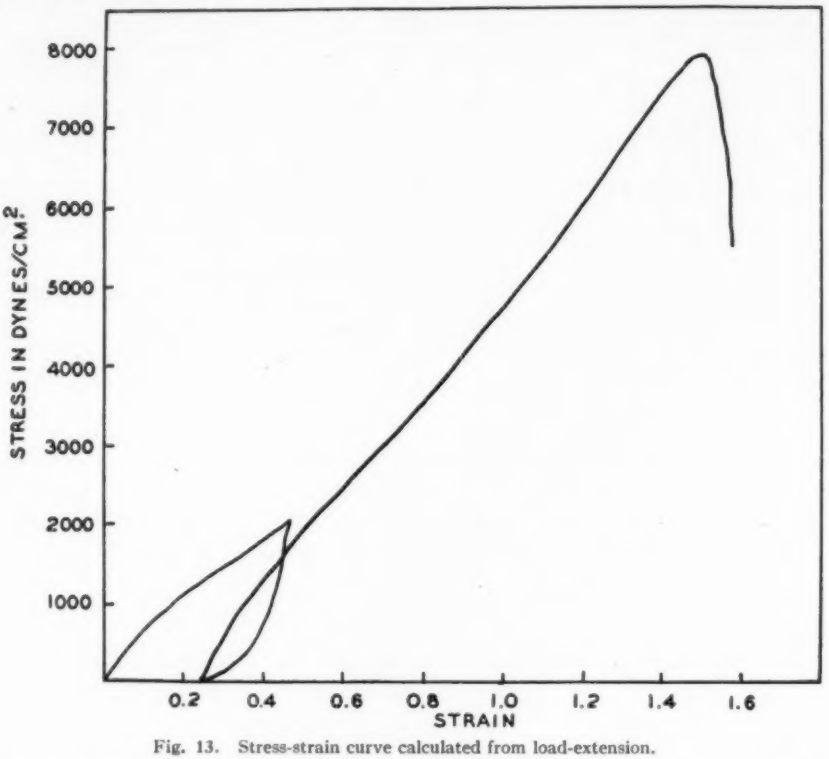

Fig. 13. Stress-strain curve calculated from load-extension.

tested, had the same extrusion time, it was found that the product figure was constant and independent of fermentation time. The results of these experiments indicate, therefore, that fermentation does not alter the strength of a flour.

The slope of the stress-strain curve is determined by the load built up during stretching. It is thus proportional to the height of the extensometer curve and is a measure of the relaxation time of the dough. Fermentation and oxidation increased the slope while reduction decreased it.

The stress-strain curve shown in Fig. 13 bends upwards slightly. With strong flours this upward bend may be quite marked, particularly when the dough is well fermented or treated with improvers. With some weak flours a downwards bend may occur. The corresponding extensometer curves are shown in Fig. 14. The increase in load with increasing extension suggests an abnormal degree of work hardening

which increases the tensile strength of the dough. The opposite effect may be due to diminishing work hardening or to the effect of structural viscosity, i.e., the fall in viscosity with increasing stress. The result is a lowering of the tensile stress.

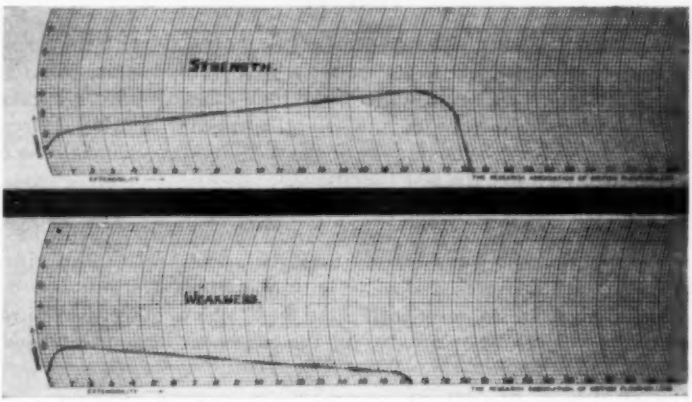

Fig. 14. Effect of strength and weakness of dough on shape of extensometer curve.

It will be realized that for any given slope of the stress-strain curve, i.e., for any given relaxation time, the greater the maximum stress the greater will be the maximum strain. In other words, the greater the tensile strength of the dough the greater will be the extensibility for a given relaxation time. Thus when properly conditioned for baking, i.e., by fermentation and improver treatment, doughs made from strong flours will stretch to a greater extent before tearing than those made from weak flours and thus should yield bread of greater volume.

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EFFECT ON DOGS OF FLOURS TREATED WITH VARIOUS IMPROVING AGENTS [1]

AARON ARNOLD [2]

ABSTRACT

Dogs were fed diets with high levels of flours treated with the following agents: ammonium persulfate (15 g./cwt.), potassium bromate (5 g./cwt.), chlorine (20 g./cwt.), benzoyl peroxide (0.8 g./cwt.), nitrosyl chloride (80 g./cwt.), chlorine dioxide (5 g./cwt.), and nitrogen trichloride (5 g./cwt.). The treatments given the flours were above those used commercially so as to increase the severity of the test conditions.

In cross-over type tests, running fits were observed only in the dogs fed nitrogen trichloride treated flour. No ill effects were seen in the dogs fed untreated flour or flours treated with the agents, other than nitrogen trichloride, given above used alone or in combination.

As judged from the tests with these dogs, agents other than nitrogen trichloride appear to be acceptable for the treatment of flour intended for human consumption.

Mellanby (3) has reported recently that dogs develop running fits when fed flour treated with nitrogen trichloride, heretofore used widely for the rapid chemical maturing of flour. Dogs appear to be somewhat more sensitive to flour so treated than other experimental animals, though untoward manifestations have been produced in ferrets (4), cats (6, 7, 8), and rabbits (7). The results in rats and monkeys appear to be equivocal (6, 7, 8), while those in guinea pigs (7) and humans (6) are negative at this writing.

These findings render the continued use of nitrogen trichloride for the maturing of flour objectionable and direct attention to alternative compounds which may be used for this purpose. Accordingly, flours treated with other agents, alone and in combination, have been fed to dogs to determine whether flour so treated would cause any untoward symptoms.

Experimental

Flour Treatments. Several fairly large experimental batches of flour were prepared in the course of the study, but due to the fact that the program extended over approximately a 2-year period, it was not feasible to apply all the various agents to the same lot of flour. Further, the length of the experiment was not anticipated fully so that each agent had to be used on more than one lot of flour. In general, however, all dogs received clear grade flour during the first 3 months of the experimental feeding period and standard patent flour during the rest

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of the experiment. The chemical agents and the levels of treatment used, together with an approximate indication of the relation of the treatment to commercial usage, are given in the first three columns of Table I.

TABLE I

TREATMENT OF FLOUR SAMPLES USED AND SUMMARY OF DOG FEEDING TRIALS

Chemical agent	Treatment level	Approximate relation to commercial treatment level	Time fed to dogs	No. of trials	No. of trials terminated by running fits
	g. per cwt. (45 kg.)	*x coml. level*	*days*		
Nitrogen trichloride[1]	5	2½–5	3–56	35	35[5]
Ammonium persulfate[2,3]	15	10	14–90	14	None
Ammonium persulfate[3] Chlorine Potassium bromate	15 20 5	10	26–42	4	None
Ammonium persulfate[3] Chlorine Potassium bromate Benzoyl peroxide[4]	15 20 5 0.8	10	21–38	10	None
Nitrosyl chloride[3]	80	10	23–38	10	None
Chlorine dioxide[1]	5	2½–5	16–28	5	None
None	—	—	8–14	4	None

[1] These samples were generously supplied by a nearby mill.
[2] In combination with inert diluents.
[3] These treated flour samples were prepared in the laboratory. The nitrosyl chloride for these studies was prepared from nitric and hydrochloric acids. It was accompanied by some chlorine.
[4] Supplied by Oxylite, available from Winthrop-Stearns, Rensselaer, New York.
[5] Average time to develop the first observed symptoms was 12 days with a range of 3 to 56 days.

It will be noted that the treatment in each case was substantially above the level ordinarily used commercially. The high level of treatment along with the high content of flour in the diets of the dogs (approximately 80%) was chosen so that the agents studied here would be subjected to a severe test.

Dogs. Young dogs were obtained for these studies. On receipt, they were dewormed and treated with distemper vaccine. They were placed on experiment when they were fully recovered from these treatments.

Diets. To test the treated flours the dogs were fed diets patterned after those of Mellanby (3). Six days a week, each dog was offered 100–200 g. of flour (steamed 90 minutes before feeding) to which was added yeast [3] (5% of weight of flour), 20 g. skim milk powder, 10 ml.

[3] Dried debittered brewers' yeast, Red Label. Vitamin Food Company, Newark, New Jersey.

corn oil, 2 g. sodium chloride, 2 g. liver concentrate powder, and 15 g. lean meat. Fish liver oil, 850 U.S.P. units of vitamin A and 100 U.S.P. units of vitamin D per ml., was given once weekly, 5 ml. per dog. In addition, each dog was given an oral supplement of 1 mg. thiamine hydrochloride, 1 mg. pyridoxine hydrochloride, 2 mg. riboflavin, 10 mg. niacinamide, 10 mg. calcium pantothenate, and 100 mg. choline chloride once weekly. The dogs had free access to drinking water at all times.

Results

The results of the dog feeding trials are shown graphically in Fig. 1. Without exception, the dogs exhibited symptoms of running fits when fed the diets which contained the nitrogen trichloride treated flour.

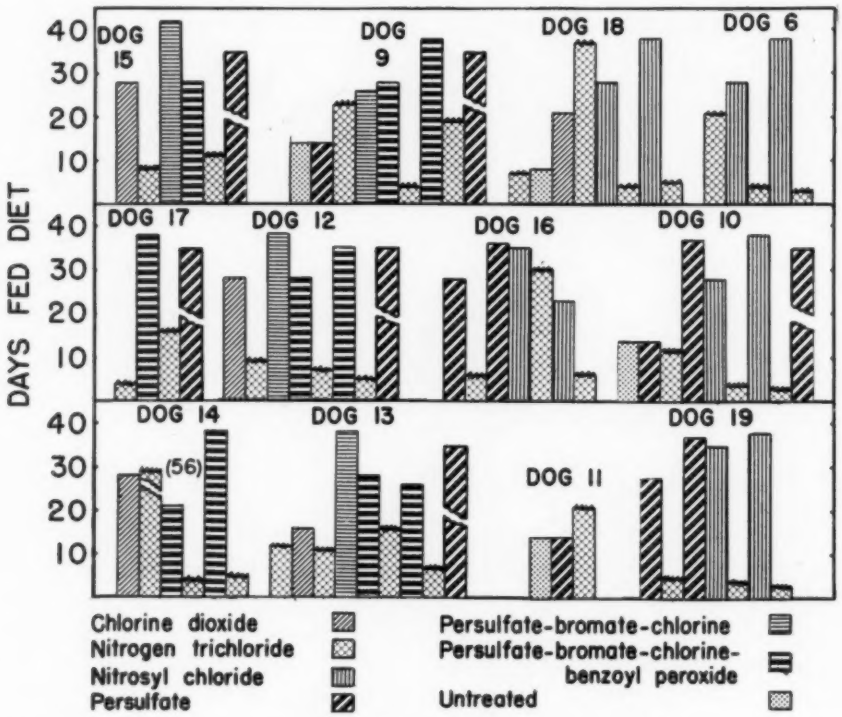

Fig. 1. Schematic representation indicating the number of days each dog was fed each diet before transfer to another diet. The development of running fits is indicated by the columns terminating in a jagged line. The interrupted persulfate columns indicate a 90-day feeding trial.

The time required for this varied and the heights of the bars show how long it took for the condition to develop to some degree in the dogs. Several factors probably operated to affect the rate of development of running fits. Some dogs reacted to being fed nitrogen trichloride treated flour by eating less; in general, these dogs developed running

fits more slowly, going for prolonged periods without symptoms, as in the cases of dogs 14, 16, and 18 (Fig. 1). The cumulative nature of the toxic principle, however, eventually precipitated the symptoms even in dogs which appeared less sensitive at times. It should be stated that, as far as possible, the dogs were transferred from the toxic diets at the earliest positive sign of the condition, for it is difficult to save a dog after a severe and prolonged attack. Some dogs were lost in spite of the attempts to prevent the development of severe symptoms.

The symptoms observed coincided with the classical description of running fits; they appeared identical with those seen earlier (1) in dogs fed a heat-processed dog biscuit of wheat flour and meat scraps. Two veterinarians associated with these laboratories concurred in the diagnosis of running fits.

No untoward symptoms were observed in the dogs fed diets which contained flour treated with other agents. The relatively high levels of treatment of the flours (2½ to 10, or more, times the commercial level) had no deleterious effect. The dogs fed the untreated flour remained equally symptom-free.

It will be noted also that flours treated with other agents not only failed to cause symptoms but were curative for dogs showing running fits as the result of ingesting nitrogen trichloride treated flour. It will be seen from Fig. 1 that on 28 occasions dogs with symptoms of running fits were transferred to other diets from the toxic diets. Thus the dogs recovered in nine instances when fed persulfate treated flour, in four instances when fed persulfate-bromate-chlorine treated flour, in six instances when fed persulfate-bromate-chlorine-benzoyl peroxide treated flour, in seven instances when fed nitrosyl chloride treated flour, in one instance when fed chlorine dioxide treated flour, and finally in one instance when fed untreated flour. That this was not due to the development of an insensitivity to the toxic agent is shown by the development of running fits when the dogs again were transferred to the toxic diet. Six of the dogs (dogs 9, 10, 12, 13, 15, 17) were fed persulfate treated flour for 90 days without exhibiting any untoward effects. These dogs were continued on test, along with five previously untreated animals, on diets which contained flour with 68 or 454 g. persulfate per 100 lbs. The results at the higher levels will be reported on at a later time.

The results are summarized in Table I. The summary results include one dog, not shown in Fig. 1, which was lost after 13 days on the toxic diet. The data on the weight records and food intakes of the dogs are not included. In spite of the high flour contents of the diets the dogs grew and gained weight steadily when fed the nontoxic flours.

Discussion

The foregoing results confirm Mellanby's findings (3) that feeding dogs flour which had been treated with nitrogen trichloride causes them to develop running fits. These findings are in accord with the earlier reports of Melnick and Cowgill (5), and of Arnold and Elvehjem (1), which indicated the nutritional origin of the running fits syndrome. This conclusion does not exclude the possibility, however, that a similar symptom complex may have other causes. For example, Weast, Groody, and Morgan (9) observed running fits in two dogs fed diets which contained heat treated protein (casein).

Other chemical agents that have been used in the past for the bleaching and/or maturing of flour do not appear to have similar undesirable effects in dogs. The results submitted herein may be compared with those of other investigators. Thus, flour treated with relatively large amounts of ammonium persulfate (approximately 10 times the commercial level) had no untoward effects in dogs. This is in agreement with the preliminary statement of Bentley *et al.* (2).

Furthermore, the treatment of flour by a combination of agents, ammonium persulfate, chlorine, potassium bromate, with and without benzoyl peroxide, appeared to have no unfavorable effects as judged by feeding trials with dogs. That benzoyl peroxide and chlorine tested separately have no adverse effects had been reported earlier by Newell *et al.* (6) and by Radomski *et al.* (7). Similarly, Bentley *et al.* (2) state that chlorine and potassium bromate tested separately did not produce the running fits factor in flour. Radomski *et al.* (7) also observed no untoward effects in dogs fed bromate treated flour.

Our observations that chlorine dioxide treated flours have no untoward effects when fed to dogs are in agreement with those of others (2, 6, 7).

Nitrosyl chloride, previously not reported on, also appears to produce in flour nothing having unfavorable effects in dogs.

Thus nitrogen chloride alone, of all the agents tested thus far, produces a factor in flour which, fed to dogs, causes the rapidly and progressively fatal running fits syndrome.

Acknowledgments

The writer is indebted to Dr. O. H. Siegmund and Dr. E. J. Morrison of these laboratories for examination of some of the dogs exhibiting running fits. The writer also wishes to express his appreciation to John C. Eggers and George M. Harder for their help in the care of the animals.

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HEAT OF HYDRATION OF CERTAIN WHEAT FLOURS AND GLUTEN [1]

W. G. SCHRENK,[2] A. C. ANDREWS, and H. H. KING

ABSTRACT

The heats of hydration of moisture-free samples of wheat flour and of gluten have been measured calorimetrically by the use of a Dewar type calorimeter, a thermocouple, and a potentiometer. Wheat flour has a heat of hydration of approximately 27 calories per gram. Slight variations occur between different flours, but they do not seem to be correlated with other properties of the flour. The rate of hydration of flour or gluten is increased by the addition of a wetting agent, without materially altering the magnitude of the energy released.

The heat of hydration of gluten (approximately 20 calories per gram) is less than that for flour, and the reaction proceeds much more slowly. This rate can also be increased by the addition of a wetting agent. The addition of cysteine to the water solution does not affect the rate or the magnitude of the energy of hydration of gluten.

Specific heat values obtained on flours and starches range from 0.42 to 0.48 calories per gram.

Winkler and Geddes (11) have shown that heats of hydration of flour and of starches vary slightly with the source of material. They made a number of measurements of this property on these materials, as well as of the specific heat of these substances. Daniels, Kepner, and Murdick (1) also made a few determinations of heats of hydration

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Contribution No. 358, Department of Chemistry, Kansas Agricultural Experiment Station, Manhattan, Kansas.

[2] Portion of a thesis presented as partial fulfillment of the requirements for the degree Doctor of Philosophy in chemistry at Kansas State College.

and of the specific heat of flour. The papers agree well on specific heat data and both agree as to the magnitude of the heat of hydration. Winkler and Geddes (11), however, indicate that the magnitude of the heat of hydration is greatly influenced by the residual moisture content of the sample, but they made no attempt to obtain a sample which was entirely free of moisture; rather, they determined the heat of hydration at various low moisture levels. Their results under these conditions indicate differences in the heats of hydration of starches and of flours from different sources.

Since the data contained in the papers mentioned do not give maximum values for the heat of wetting because of the moisture present, and were obtained by the use of relatively large concentrations of material, it seemed desirable to attempt an accurate measurement of heats of hydration at minimum moisture levels and in dilute mixtures of the samples and water, and thus attempt to obtain a measurement of maximum value for the energy of hydration. These data would then be available as fundamental information and for use in possible correlations with other known properties of the materials.

The energy of hydration of gluten was also considered important from the standpoint of total energy of hydration of flour, and data on several samples of gluten are included. A few specific heat measurements were also made in order to verify existing data. The effect of the presence of a wetting agent was also determined since it is known that wetting agents have an effect on mixogram patterns (7) and consequently affect baking qualities.

Materials and Methods

Preliminary experiments indicated that the imbibition of water by flour or gluten was quite rapid, and that the use of a Dewar flask type of calorimeter was adequate for the measurements of heats of hydration. The arrangement of the calorimeter and associated equipment has been previously described (6).

Temperature measurements were made by the use of a calibrated 50 junction copper-constantan thermocouple and a type K potentiometer. With the galvanometer arrangement used a change in potential of 0.001 mv. could be detected. This potential difference, together with the sensitivity of the thermocouple, gave a sensitivity of ± 0.00046°C.

Determination of the Heat of Hydration. The technique of making measurements of the energy of hydration was similar to that developed for the heats of immersion of paint pigments by Harkins and Dahlstrom (2). The method was essentially as follows: A sealed sample tube containing the sample was placed in the calorimeter immediately

above 400 to 425 g. of distilled water. The sample tube was arranged so that the bottom could be broken from outside the calorimeter and the sample allowed to fall into the water. All the contents of the calorimeter were brought to the temperature of the thermostat in which the calorimeter was immersed. After temperature equilibrium had been attained the stirrer was started and electrical connections made.

Temperature readings were then taken at one-minute intervals in order to establish the slope of the time-temperature curve. The sample was then introduced into the water by breaking the sample tube. Readings were continued and the energy released by the reaction was measured by means of the temperature rise produced. After the temperature rise had reached its maximum, readings were continued in order to re-establish the slope of the time-temperature curve. This was followed by the addition of electrical energy to the calorimeter by means of a current passing through the heater coil in the calorimeter for a known period of time. Current was measured with a potentiometer and a standard one-ohm resistance. Current drain was low and the source of current was heavy-duty storage cells. A small vernier resistance permitted precise control of current. The temperature rise produced by the electrical energy was measured as before.

Fig. 1 represents the type of curve obtained by such measurements. The solid line represents the results to be expected if the energy releases and temperature rises were instantaneous. The segment AB represents the establishment of the time-temperature curve previous to breaking the sample tube. BC represents the rise in temperature produced by the release of the energy of hydration; CE represents the new time-temperature slope; and EF represents the rise in temperature produced by the electrical energy released by the heater coil.

The electrical energy released, and represented by EF, can be calculated in calories as follows:

$Q_2 = \frac{I^2Rt}{4.1833}$ where I is the current in amperes, R is the resistance of the heater coil in ohms, and t is the time in seconds.

If Q_1 represents the energy required to produce a temperature rise equal to BC, it follows that

$Q_1 = Q_2\frac{BC}{EF}$ where Q_1 is the energy of hydration of the sample. The total rise in temperature during the course of an experiment was approximately 0.8°C. A typical set of data is shown in Fig. 2.

This method of making measurements eliminates a number of troublesome calculations, including energy equivalence of the calori-

meter, specific heat of the sample, and accurately measured amounts of water. Temperature measurements do not need to be changed to degrees, but may more conveniently be expressed in terms of millivolts. The accuracy of measurement thus depends on the accuracy of the measurements of the electrical constants and the precision of timing.

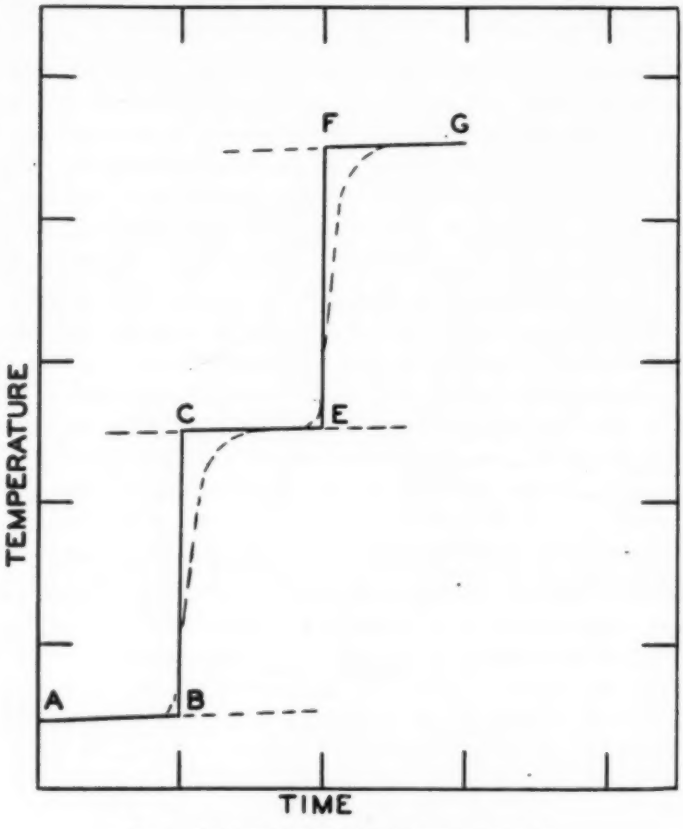

Fig. 1. Method of calculating the temperature rise.

The calorimeter and measuring equipment was checked for accuracy against the heat of neutralization of hydrochloric acid with sodium hydroxide. Excellent agreement with the data of Richards and Rowe (4) was obtained.

Preparation of Samples. Samples were prepared by drying overnight in a vacuum oven at 100°C. They were then placed in sample tubes as previously described (6) and dried under vacuum again. The tubes were sealed under vacuum. Numerous moisture determinations made on samples prepared in this way indicated that essentially no moisture remained. The tubes were placed in the calorimeter and allowed to reach temperature equilibrium with the contents after

which they were broken in such a way as to get rapid mixing of the sample and water (6). The entire calorimeter was immersed in a constant temperature water bath maintained at 25 ± 0.1°C., and all measurements were made at this temperature.

The literature on the effect of the heat treatment used in drying these samples is not entirely in agreement. Winkler and Geddes (11)

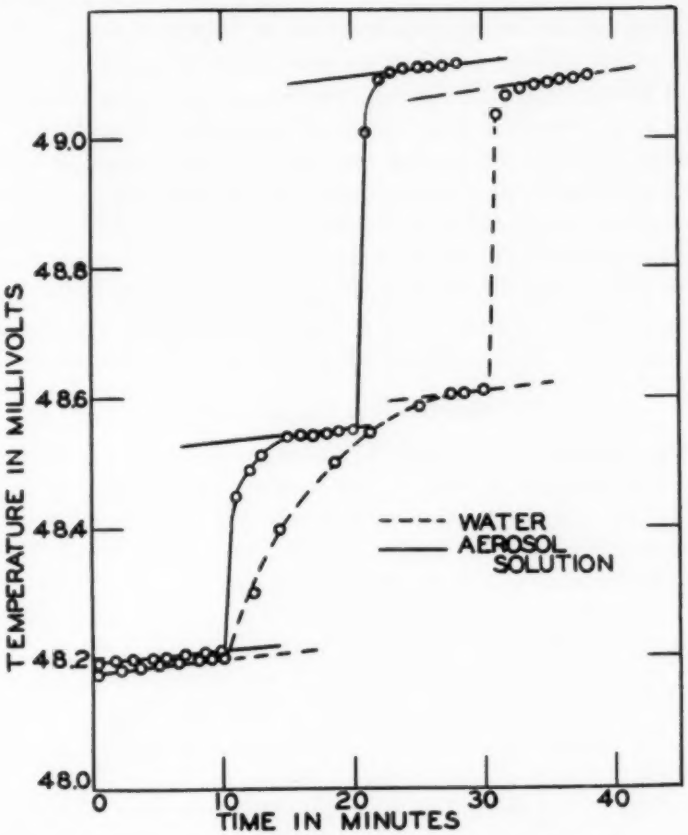

Fig. 2. Effect of aerosol OT on the time-temperature curve of gluten.

have shown that heat treatment affects the hydration reaction slightly. Sair and Fetzer (5) also reported that the sorption capacity of starch is somewhat altered by heating. Hellman and Melvin (3) indicated that in a series of corn starches from different sources and treatments, there was no significant change in moisture sorption capacity. Wands (10) also has recently reported on the sorption of water by starches, in which some samples were autoclaved at high temperature and pressure. This treatment produced no measurable change in the water sorption capacity of the starch. In view of the nature of the above data it

seems likely that the heat treatment accorded these samples produced relatively little effect. Furthermore, since all samples were accorded the same treatment, any slight changes produced would be similar and comparisons would remain valid. It is very likely that any remaining traces of moisture would have a much greater influence on the data than did the heat treatment.

Determination of Specific Heat. Specific heat calculations require precise data on the energy equivalence of the calorimeter. This was determined by placing a known quantity of water in the calorimeter, adding a known amount of electrical energy, and calculating the temperature rise. Specific heat measurements were then made in the same manner with the sample in place. The sample was packed tightly into a long, thin-walled brass tube which was then immersed in a known quantity of water in the calorimeter. Time was allowed to permit the calorimeter and its contents to reach temperature equilibrium at 25°C. Electrical energy was then added as before and the rise in temperature calculated. From these data the specific heat of the sample could be calculated since all other factors were known.

Results and Discussion

Heat of Hydration of Flour. The heats of hydration determined on a number of different samples of wheat flour vary over only a narrow range (Table I), except for sample 3 which gave a very high value. This discrepancy may be due to the fact that this sample had been bleached with chlorine, the only flour in the series which was so treated.

The time required for the hydration reaction to reach completion was also quite constant. An average time of about 9 minutes was required. This is considerably longer than for starch alone, which averaged about 4 minutes. Gluten, in contrast, required about 17 minutes. Evidently the reaction time for flour is influenced considerably by the presence of the gluten in the flour.

The data in columns 2, 3, and 4 of Table I were obtained from measurements made with the use of the recording dough mixer (9). These properties do not present any evident correlation with the heat of hydration values. Tenmarq and Chiefkan flour, which are quite different in baking properties, also do not show measurable differences in their heats of hydration.

Hydration of Gluten. The data in Table II present information on the energy of hydration of gluten. Gluten releases its energy of hydration much more slowly than starch or flour. Grinding of the gluten made no difference in the time required for hydration. The energy of hydration of the gluten is considerably below that of flour and starch, averaging about 20 calories per gram.

TABLE I

COMPARISON OF THE HEAT OF HYDRATION OF FLOURS WITH CERTAIN OTHER PROPERTIES OF FLOURS AND THEIR DOUGHS [1]

Sample	Nature of flour	Protein[2]	Mixogram curve measurements			
			Mixogram angle at peak	Area under curve	Height	Heat of hydration
		%	*degrees*	*cm.*^{2}	*cm.*	*cal./g.*
1	Cake	9.4	6	33.7	2.2	27.6
2	Cake	8.8	4	39.4	2.9	27.2
3	Cake (Cl_2 bleach)	12.0	19	72.8	6.2	30.0
4	Bakery	11.7	25	71.4	6.0	27.2
5	Bakery	12.9	12	79.4	5.8	26.8
6	Cracker dough	9.4	7	38.1	2.8	27.3
7	Cracker dough	9.5	3	42.9	3.0	26.4
8	Cracker sponge	10.8	25	61.4	5.6	27.5
9	Cracker sponge	10.9	10	73.0	5.0	25.2
10	Family	11.6	10	74.1	5.4	26.8
11	Family	10.2	14	64.0	5.7	26.2
12	Cooky flour	8.1	2	37.2	2.2	26.0
13	Cooky flour	10.8	7	63.7	4.7	27.2
14	White bread	14.6	13	90.1	6.3	26.4
15	Topping	12.1	7	70.4	4.9	28.2
16	Tenmarq flour	—	—	—	—	27.0
17	Chiefkan flour	—	—	—	—	27.1

[1] All samples dried as explained in the experimental section.
[2] At zero moisture content.

TABLE II

HEAT OF HYDRATION OF GLUTENS IN WATER AND WATER SOLUTIONS [1]

Source of gluten	Treatment	Time for hydration	Heat of hydration
		min.	*cal./g.*
Chiefkan wheat	None	18	20.0
Tenmarq wheat	None	18	20.2
Chiefkan wheat	1% Aerosol	6	19.8
Tenmarq wheat	1% Aerosol	6	19.5
Chiefkan wheat	0.5 g. Cysteine	19	19.8
Tenmarq wheat	0.5 g. Cysteine	18	21.0

[1] All samples dried as explained in the text.

Effect of Adding a Wetting Agent. Swanson and Andrews (7) have shown that the addition of a wetting agent markedly changes the shape of the mixogram patterns in flour and consequently affects the baking qualities of the flour. The addition of a wetting agent (1% Aerosol OT [3]) reduced the time required for the release of the energy of hydration of gluten to about 6 minutes.

[3] Obtained from American Cyanamid and Chemical Corporation.

This change in time is illustrated by the two curves of Fig. 2. The vertical heights of the lower segments of the two curves are not comparable because of different sample weights.

The magnitude of the energy released was not greatly changed. Apparently the reduction in interfacial tension produced by the addition of the wetting agent permitted the water to penetrate into the particle much more rapidly and thus allowed more rapid release of the energy of hydration without seriously affecting its magnitude. This more rapid penetration would have a marked effect on the mixogram pattern of flour regardless of the energy changes involved. An increased reaction rate for flour was also observed when the wetting agent was used for these materials.

Effect of Adding Cysteine to Gluten. Swanson and Andrews (8) have shown that the addition of cysteine to a dough produces a marked effect on mixogram patterns. The effect of cysteine on mixogram patterns is not reflected in measurements of the heat of hydration since it did not change the heat of hydration of gluten or the rate of release of energy.

Data on the effects of cysteine and aerosol OT are presented in Table II.

Specific Heat of Flour and Starch. The specific heat of flour and starch has been previously determined by Daniels *et al.* (1) and also by Winkler and Geddes (11). Since the calorimetric equipment used here offered an excellent opportunity to check these data, several experiments were performed to determine this property of flour and starch. The data obtained on several samples are given in Table III.

TABLE III

SPECIFIC HEAT OF SEVERAL STARCHES AND FLOURS

Sample	Specific heat
	Cal./g.
Wheat starch	0.48
Wheat starch	0.47
Wheat flour	0.45
Wheat flour	0.43
Waxy corn starch	0.46
Waxy corn starch	0.44
Blackhull starch (from sorghum grain)	0.45
Blackhull starch (from sorghum grain)	0.42

These data may be compared with the value of 0.397 reported by Winkler and Geddes (11), and 0.425 reported by Daniels *et al.* (1). The specific heat of the samples of wheat starch is slightly greater than that of wheat flour, waxy corn starch, and sorghum starch.

Acknowledgment

Appreciation is expressed to the Department of Milling Industry of Kansas State College and especially to Dr. J. A. Shellenberger of that department for supplying the samples used; also, for permission to use the data obtained with the recording dough mixer presented in Table I.

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THE BIOLOGICAL VALUE OF SOME COMMERCIAL CORN PROTEIN FRACTIONS [1]

J. A. SCHULZ and BYRON H. THOMAS [2]

ABSTRACT

A number of workers have emphasized recently the potential value of corn germ in the human dietary. With few exceptions the corn germ investigated to date has been produced by the dry milling of whole corn. In this study, the materials used were commercial by-products of the wet-milling of whole yellow corn. They were steep water concentrate, corn gluten, and variously processed corn germ.

A series of nitrogen metabolism trials were conducted, using groups of mature male rats, to determine the effects of method of processing and aging on the coefficient of true digestibility and biological value of certain of these nitrogen supplements.

The protein of corn germ that had been expeller processed was less efficiently utilized biologically than the same corn germ which had been solvent-extracted to remove the oil.

Solvent-extracted corn germ that had been aged at room temperature a few months, two years, and six years yielded biological values of 83 ± 1.5, 79 ± 1.9, and 55 ± 1.4, respectively, demonstrating unmistakably the deleterious effect of storage.

The feasibility of conducting the same rat through three nitrogen balance tests is discussed.

Recently, a number of authors have reported upon the protein value of corn germ, emphasizing its potential value in the human dietary. The nutritional limitations of corn and certain of its by-products were demonstrated years ago by Osborne and Mendel (15, 16). In these early studies, growth response made by rats was the index of quality. Later Mitchell (9, 10), using a less empirical procedure, found the biological value of the proteins of whole corn to be 72 and 60 when included in the ration at levels of 5 and 10%, respectively.

Mitchell and Beadles (12), utilizing the Mitchell nitrogen balance method, found the biological value of the protein of dry-milled solvent-extracted corn germ to be 77.6, which was equal to that of round of beef and 85% as digestible. Jones and Widness (6) compared the growth-promoting values of wheat germ and corn germ proteins with those of various other vegetable and animal proteins by feeding them to weanling rats at protein levels of 10, 15, and 17.5% of the ration. At all of these levels, they found wheat germ protein to be superior to corn germ protein.

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[2] Research Assistant Professor and Research Professor Animal Chemistry and Nutrition Subsection.

Recently Beeson, Lehrer, and Woods (1) reported upon the protein values of wheat germ and corn germ using the growth-promoting method with young rats. As sole source of protein in the ration, both wheat germ and corn germ compared favorably with whole egg protein in the rate of gain made by the rats, but the corn germ was less efficient. As a co-supplement with peas, corn germ protein fed at various percentage levels also was somewhat less efficient than wheat germ under the conditions of their experiments.

Stare and Hegsted (17) confirmed the relative superiority of wheat germ compared to corn germ as a supplement to wheat flour, but found that for maintaining the nitrogen balance in the adult dog, the proteins of skim milk powder, wheat germ, and corn germ were practically of equal value.

Hove and Harrel (4) reported corn oil meal (corn germ) to have a protein value of 2.40, which represented grams gain in weight made by rats per gram of protein eaten. On the same basis, they found corn oil meal to have a protein value of 1.71 as a supplement to white patent flour, compared to a protein value of 2.26 for both dry skim milk and defatted wheat germ. Soybean oil meal on this basis had a protein value of 1.77. Block and Bolling (2) reported the protein value of solvent-extracted corn germ to be 1.8 while corn germ extracted by the "hot expulsion method" had a protein value of 2.1.

With few exceptions the corn germ investigated to date has been produced by the dry milling of whole corn. In this study, the by-products used were commercial products of the wet-milling of whole yellow corn. By far the largest percentage of commercial corn proteins on the market is produced by this method.

Materials and Methods

This test comprised a series of nitrogen metabolism trials in which groups of mature male rats six months old and weighing from 250 to 350 g. at the start of the experiments were used to determine the coefficient of true digestibility and biological value of various protein by-products of the corn milling industry. The supplements tested and pertinent remarks concerning their preparation are given in Table I. Brief reports concerning these have appeared in the annual reports of this station (3).

Each supplement was fed to groups of 11 rats. Uniform environmental conditions were maintained for all rats throughout the period of each test and insofar as possible between tests. Each test material was included in a diet which we believe was otherwise nutritionally complete for mature rats and was fed *ad libitum* for a period of 14 days to rats caged separately. Each rat was conditioned to its test ration

during the first 7 days of this period. A 14-day low-nitrogen period, during which the rats received only the basal diet, both preceded and followed each test nitrogen (nitrogen assay) period. Data pertinent to the determination of coefficients of these digestibility and biological values were collected during the last 7 days of each 14-day period.

TABLE I

PROTEIN SUPPLEMENTS USED AND THEIR METHOD OF PREPARATION[1]

Expt. no.	Supplement	Percentage protein[2]	Remarks
1	Corn steep water concentrate	27.10	Contains acid and water-soluble portions removed by steeping the corn and washing the starch.
2	Corn germ[3]	20.75	Oil removed by laboratory extraction with diethyl ether.
8	Corn germ[3]	20.75	Oil removed by laboratory extraction with diethyl ether, then aged for two years at room temperature (70°–90°F.) in metal container.
20	Corn germ[3]	20.75	Oil removed by laboratory extraction with diethyl ether, then aged at room temperature for six years. Had acquired a musty and somewhat rancid odor.
3	Corn germ	25.30	Oil removed commercially by expeller process.
4	Corn gluten	44.00	Standard commercial product.
5	Soybean oil meal	44.46	Oil removed commercially by solvent extraction. Meal had aged several years prior to its use experimentally.

[1] All supplements except soybean oil meal are products of commercial wet-milling process. Samples utilized in Experiments 1–4 inclusive were supplied by the American Maize Products Company through the courtesy of H. H. Schopmeyer.
[2] Protein = N × 6.25.
[3] Represent same corn germ aged differently as noted.

The basal, low-protein ration had the following percentage composition: starch 86, filtered butterfat 8, McCollum's salts No. 185 (7) 4, and agar-agar or celluration (Fisher) 2%. The protein supplements to be tested replaced equal parts by weight of the starch so that the test rations contained approximately 5% crude protein. The actual percentage of protein in each ration was determined chemically. Each rat received in addition 50 mg. of yeast concentrate (Harris) and 20 μg. thiamine chloride daily, and three drops of a cod liver oil concentrate (Upjohn) weekly.

The ration for each rat was weighed out daily. It was placed in feeding devices so constructed that spillage was practically eliminated. Uneaten feed was weighed back each morning. Whenever feed was spilled, it was added to the next day's ration so that each rat actually ingested all the feed with which it was credited. Each rat was offered daily all the feed it could consume. Distilled water was fed *ad libitum*.

Feces and urine were collected quantitatively and preserved for analysis each day during the last 7 days of the period. The urine, which was absorbed on acidulated filter paper, and the feces were predigested with 20% hydrochloric acid solution. At the close of the 7-day collection period, aliquots of the digests of both total urine and feces were further digested with sulfuric acid. Nitrogen was determined by the Kjeldahl method using copper sulfate as a catalyst. Except for minor modifications, the method used for determining the coefficients of true digestibility and biological value of the proteins was that developed by Mitchell and associates (9, 10, 13).

Results

The extent to which rats digested or utilized the proteins in steep-water concentrate, variously processed corn germ, and corn gluten is reported in Table II. All of these supplements were highly digestible.

TABLE II

UTILIZATION OF PROTEIN FROM DIFFERENT SUPPLEMENTS BY MATURE RATS

Supplement	Expt. no.	Rat nos.	Percentage protein[1]		Coeff. of true digestibility[2]	Biol. value[2]	Avg. wt. change of rats[2]
			Supplement	Ration			
			%	%	*Avg.*	*Avg.*	*g.*
Steep water concentrate (from corn)	1	1–11[3]	27.10	6.19	88±1.0	35±2.6	−5.8
Corn germ (commercial) ether extracted[4]	2	1–11	20.75	5.37	72±0.9	83±1.5	+6.0
Corn germ (commercial expeller process)[4]	3	12–22	25.30	5.18	70±1.0	66±1.2	+1.6
Corn gluten (commercial)	4	12–22[5]	44.00	5.36	89±0.7	68±2.6	−9.5
Soybean oil meal (solvent extracted)	5	12–22	44.46	5.49	83±0.9	67±1.5	−5.9

[1] Protein = N × 6.25.
[2] Computations based on a 7-day collection period following a conditioning interval of similar length. ± = probable error.
[3] Same rats used in Experiments 1 and 2, and in 3, 4, and 5.
[4] Both samples of germ derived from same batch of whole corn.
[5] One of 11 starting rats "off feed" during test. Calculations are mean values based on data from remaining rats.

The biological values of their proteins differed greatly. As anticipated, steep water concentrate had a biological value of only 35. On the other hand, solvent-extracted wet-milled corn germ (Experiment 2) gave a biological value of 83. This value would seem to compare favorably that with of 77.6 obtained for dry-milled solvent-extracted corn germ by Mitchell and Beadles (12), even though our corn germ was fed at a 5% level to mature rats, and theirs at a 10% level to growing rats. Stare and Hegsted (17) state that the wet-milling process probably destroys some of the nutritive value of corn germ.

If this is so, it would seem as though the magnitude of destruction is small judging from the results presented here.

Recently, Mitchell, Hamilton, and Beadles (14), while demonstrating that the biological value and digestibility of the proteins of soybeans could be improved by mild heat-processing, commented upon the sensitivity of the cereal proteins to heat and warned against some of the methods used in their preparation. The correctness of this statement seems to be borne out by the results of Experiments 2 and 3 reported in Table II. Both samples of corn germ were derived from the same batch of whole corn and both were commercially wet-milled. That used in Experiment 2 was subsequently laboratory-extracted with diethyl ether, whereas the germ used in Experiment 3 had been expeller-processed. Both samples of germ were equally digestible; however, the expeller-processed germ had a biological value of only 66 as compared to 83 for the germ solvent-extracted in the cold. The difference in these results very probably indicates the effect of heat treatment during expeller-processing upon the native proteins of the corn germ. The biological value of 68 established in Experiment 4 for corn gluten demonstrates that the proteins of this supplement were utilized as well as those of the expeller-processed corn germ (Experiment 3).

TABLE III

EFFECT OF LENGTH OF STORAGE AT ROOM TEMPERATURE OF A SAMPLE OF ETHER-EXTRACTED CORN GERM UPON THE UTILIZATION OF ITS PROTEIN BY MATURE RATS

Duration of storage	Expt. no.	Rat nos.	Percentage protein[1]		Coeff. of true digestibility[2]	Biol. value[2]	Avg. wt. change of rats[2]
			Corn germ	Ration			
Years					*Avg.*	*Avg.*	*g.*
¼	2	1–11	20.75	5.37	72±0.9	83±1.5	+6.0
2	8	23–33	20.75	5.02	81±1.0	79±1.9	+3.8
6	20	69–79[3]	20.75	5.09	76±0.8	55±1.4	−3.1

[1] Protein = N × 6.25.
[2] Computations based on a 7-day collection period following a conditioning interval of similar length. ± = probable error.
[3] Four of 11 rats started refused to eat ration. Calculations are mean values based on data from remaining rats.

Jones, Divine, and Gersdorff (5) and Mitchell (11) have reported that the storage of either whole or ground corn for periods of several months or longer resulted in a decrease in the biological value of the proteins of the corn. The data presented in Table III show the effect of storage upon the biological value of the ether-extracted sample of commercial corn germ used originally in Experiment 2. Note that this sample showed a biological value of 83 and a coefficient of true digesti-

bility of 72 (Experiment 2). It was stored for 6 years at room temperature and biologically tested when 2 and 6 years old. The harmful effect of storage on the ether-extracted germ was moderate after 2 years, but very pronounced after 6 years. The coefficient of true digestibility had, however, risen from 72 to 81 after 2 years' storage, which is a significant increase.

The corn germ which had been stored for 6 years at room temperature had a musty, somewhat rancid odor so that at the beginning of the 14-day test period of Experiment 20, only a few of the experimental rats ate liberal amounts of the ration into which it was corporated. However, by the time the preliminary 7-day period was completed, seven of the 11 rats were eating the diet satisfactorily. These seven rats established a biological value of 55 for the 6-year-old germ in marked contrast to 83 for the same germ comparatively unaged. The coefficient of true digestibility of 76 for the 6-year-old germ was between the value of 72 for the fresh sample and 81 for the 2-year-old sample. No explanation for these moderate shifts in the digestibility of this germ can be offered at this time; however, the effect of the lengthy storage upon the biological value of the proteins is unmistakable.

Discussion

There has been some question whether the same group of rats should be conducted through more than two protein test periods. Rats no. 12–22 after having been subjected to two 14-day protein test periods and three 14-day low-protein periods, although somewhat attenuated, established in Experiment 5 an average biological value of 67 for a sample of solvent-extracted soybean oil meal. This value compared favorably with the figure of 67.5 obtained with the rats used by Mitchell and Beadles (12). Mitchell and Beadles used young rats and fed autoclaved soybeans at a protein level of 10% of the ration. Melnick, Oser, and Weiss (8) obtained a biological value of 71 for autoclaved soybeans when fed at a protein level of approximately 9% of the ration. It would appear that the biological value of 67 (Experiment 5) is not in disagreement with the values obtained by others when it is considered that our sample of solvent-extracted soybean oil meal possibly had been heated insufficiently (8), had been stored at room temperature for several years (10), and was fed at a 5% protein level. Rats 23–33 (Table III, Experiment 8) established a biological value of 79 for corn germ stored 2 years, after satisfactorily establishing two previous biological values for as many different supplements; thus again indicating that it is feasible to conduct the same rats through three protein test periods.

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THE BIOLOGICAL VALUE OF THE PROTEINS OF CORN GERM AND ENDOSPERM WET-MILLED EXPERIMENTALLY [1]

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ABSTRACT

The coefficients of true digestibility and biological value have been determined, using conventional procedures, primarily for samples of germ and endosperm produced by the experimental wet milling of certain inbred lines and a related hybrid of corn. The inbred lines tested were OS426 waxy and starchy and 1205 waxy and starchy; the hybrid, Iowa 939 waxy.

No highly significant differences were found between the biological values of the germ and endosperm proteins from the waxy and starchy inbreds, nor between these and comparable protein fractions from the hybrid Iowa 939 waxy. It is recognized that more marked differences in biological values might have been obtained had the genetic lines used been more unrelated.

None of the proteins used in this study produced the high biological values exhibited by a commercial wet-milled sample of corn germ reported in a previous paper. This may be due to the fact that the commercial nitrogenous by-products used in the previous study were derived from corn of more heterogeneous origin while the present samples were from Iowa 939 and its single-cross inbred parents. These differences may also be due to variations in the commercial and laboratory technique used in wet milling, resulting in somewhat differently constituted end products.

It has long been known that the percentage of crude protein in the corn kernel could be influenced by proper management and fertilization of the soil in which the corn was grown. Very recently, Richter (9) increased the crude protein of Iowa 939 corn from 8.8 to 9.4% by fertilization of the soil. Whether the biological values of the corn proteins were influenced by such cultural procedures is not known. It is well established that the percentage of crude protein can be influenced by the genetic constitution of the corn. Certain corn hybrids differ markedly in the percentage of crude protein they contain, and there is some evidence that the biological values of the proteins of such hybrids also may differ. However, accumulated evidence bearing on the latter is still fragmentary.

Kik (3) found that various varieties of rice differed in the biological value of their proteins. Marais and Smuts (8) reported the biological value of "whole white maise" to be 76 $\pm$ 1.91 while that of "whole yellow maise" was 67 $\pm$ 0.98. Doty, Bergdoll, Nash, and Brunson (1) showed that "the amounts of cystine, arginine, histidine, tryptophan, and tyrosine present in two samples of single-cross hybrid corn

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differed considerably as did the percentages of alcohol and alkali soluble nitrogen."

The high biological value of 83 obtained with a sample of commercial wet-milled corn germ in 1940 (2), and the discovery in 1942 (2) that the protein of the germ from a sample of laboratory wet-milled Iowa 939 waxy corn had a much lower biological value, namely 66, raised the question whether or not this lower figure resulted from genetic characteristics inherent in the waxy strain of Iowa 939. At that time, Iowa 939, whose starch had been found to be an acceptable substitute for tapioca starch, had just been developed. Limited amounts of corn from the inbred lines of OS426 waxy and starchy, and I205 waxy and starchy employed in the production of the hybrids Iowa 939 waxy and Iowa 939 nonwaxy became available. The coefficients of true digestibility and biological values of certain nitrogenous by-products derived from this genetic material constitute the basis of this report.

Materials and Methods

The samples of corn referred to above were experimentally wet-milled.[3] Samples of diethyl ether-extracted corn germ and of the corn endosperm were made available. Also provided for study was a sample of OS426 starchy corn endosperm which had been extracted with ethanol to remove most of the zein. The coefficients of true digestibility and biological values were determined by the same procedure as outlined in a companion article (10). Briefly, each test material was fed individually to groups of 11 mature male rats. All rats were conditioned during the first 7 days of a 14-day test period. Data concerning feed eaten and urine and feces elimination were collected quantitatively during the last 7 days and appropriately preserved for chemical and statistical analysis.

The test materials were included at the 5% level in a basal ration (10) nutritionally complete for rats except for the protein. The ration for each rat was weighed out daily, and placed in feeding devices so constructed that spillage was almost completely eliminated. Any spilled or unconsumed feed was accounted for quantitatively.

Except for minor modifications, the procedure used in determining biological value and coefficient of true digestibility of the test materials was that developed by Mitchell and associates (4, 5), as specified in the preceding paper (10).

[3] The samples of inbred lines of corn used in this project were furnished us by courtesy of Dr. G. F. Sprague, Senior Agronomist, U. S. D. A., Ames, Iowa. They were grown on test plots during the same season under comparable conditions and processed at the same time so that possible differences in soil fertility, available moisture, stage of maturity, storage, etc., and their effects on the quality of the proteins should not be a consideration here.

The whole corn samples were wet-milled by the Plant Chemistry Subsection of the Iowa Agricultural Experiment Station. The endosperm samples contained all the endosperm of the corn while the germ samples were as free from adhering endosperm as was possible with the experimental method of wet milling employed.

Results

Data concerning the quality of the protein and its digestibility in each of the samples of corn germ tested are reported in Table I.

TABLE I

UTILIZATION OF PROTEIN FROM CORN GERM OF DIFFERENT GENETIC CONSTITUTION AS MEASURED BY MATURE RATS

Source of germ	Expt. no.	Rat nos.	Percentage protein[1]		Coeff. of true digestibility[2]	Biol. value[2]	Avg. wt. change of rats[2]
			Germ	Ration			
			%	%	*Avg.*	*Avg.*	*g.*
OS426 waxy[3]	10	34–45[4]	25.03	5.11	83±1.1	72±1.4	+0.5
OS426 starchy	9	34–44[4]	25.63	4.94	82±1.0	72±1.6	+1.7
I205 waxy	11	34–45[4]	26.37	5.11	85±0.5	67±1.4	+0.6
I205 starchy	12	46–56	23.91	5.38	79±0.5	64±1.7	−7.4
I205 waxy I205 starchy OS426 waxy OS426 starchy (equal parts of each)	13	46–56	—	5.07	81±0.6	65±1.3	+2.7
Iowa 939 waxy	7	23–33	24.27	5.02	83±0.6	66±1.2	−5.4

[1] Protein = N × 6.25.
[2] Computations based on a 7-day collection period following a conditioning interval of similar length. ± = probable error.
[3] Corn from inbred lines used in this study obtained by courtesy of G. F. Sprague, Senior Agronomist, U. S. D. A., Iowa State College, Ames, Iowa. All samples were wet-milled in the laboratories of the Plant Chemistry Subsection of the Iowa Agricultural Experiment Station.
[4] Rat no. 35 became sick during Experiment 9 and was discarded. Replaced by no. 45 in Experiments 10 and 11.

Apparently there are no highly significant differences between the average biological values of the various waxy and starchy inbreds, nor between these and a composite of them, or hybrid Iowa 939 waxy. The largest differences in average biological values are between those of OS426 waxy or starchy and the following: the I205 inbreds; a composite of all inbreds; and Iowa 939 waxy. It is quite probable that these differences are more apparent than real. If the differences in these biological values were real, then the biological value of the composite of corn germ (Experiment 13) should have been somewhat higher than 65 ± 1.3. Differences in the rats used and their day-to-day condition, not always noted nor always shown by the probable errors of their biological values, frequently make differences of this magnitude nonsignificant. There were no significant differences between the average biological values of the corn germ proteins of the starchy and waxy inbreds in each line, namely, OS426 and I205.

Similarly, the data in Table II show that there were no significant differences between the average biological values of the corn endosperm proteins of OS426 and I205 whether one considers either the waxy or starchy varieties. These biological values, namely, 54, 59, 55, and

55, are so much alike that they might well have all been determined upon the same sample. This similarity in the nutritive value of these endosperm proteins serves to emphasize further the contention that the differences in the biological values obtained from the corn germ proteins of these inbred lines were more apparent than real.

TABLE II

UTILIZATION OF PROTEIN FROM CORN ENDOSPERM OF DIFFERENT GENETIC CONSTITUTION AS MEASURED BY MATURE RATS

Source of endosperm	Expt. no.	Rat nos.	Percentage protein[1]		Coeff. of true digestibility[2]	Biol. value[2]	Avg. wt. change of rats[2]
			Endosperm	Ration			
			%	%	*Avg.*	*Avg.*	*g.*
OS426 waxy	17	57–68[3]	10.55	5.13	94±1.3	54±2.4	− 7.3
OS426 starchy	15	57–67	10.75	5.17	94±1.4	59±1.7	− 4.6
I205 waxy	18	69–79	11.34	5.02	91±1.1	55±1.3	−10.4
I205 starchy	19	69–79[4]	10.88	5.15	93±1.1	55±2.1	− 8.8
OS426 starchy[5]	16	57–67	4.08	3.60	89±1.2	70±1.7	− 3.6
Corn gluten (Iowa 939 waxy)	6	23–33	42.22	5.23	95±0.6	44±1.9	− 9.2

[1] Protein = N × 6.25.
[2] Computations based on a 7-day collection period following a conditioning interval of similar length. ± = probable error.
[3] Rats no. 59 and 60 became sick during Experiment 17. Rat no. 60 replaced by no. 68.
[4] Rat no. 76 became sick. Discarded.
[5] Ethanol extracted to remove most of zein.

The differences between the biological values of the samples of corn endosperm and the biological value of 44 ± 1.9 obtained for Iowa 939 waxy gluten (Experiment 6) are highly significant. The more starchy corn endosperm proteins constitute the original source of Iowa 939 waxy gluten utilized in Experiment 6. The endosperm sample contained much starch and it may be that corn gluten when encapsulated with its normal starch component has a somewhat higher biological value than when the starch is largely removed. Also, it is possible that in the removal of the starch from the gluten small amounts of more nutritionally complete protein are removed. The early work of Osborne and Mendel (6, 7) demonstrating the relative values of zein, corn gluten, and the corn glutelins would make such an explanation plausible. Finally, it is possible that in the process of separating the corn gluten from the starch, the gluten undergoes some degree of protein denaturation.

A sample of OS426 starchy endosperm which had been extracted with ethanol analyzed only 4.08% crude protein. Without further removal of carbohydrate it was impossible to introduce this ethanol-extracted endosperm into the basal ration in sufficient quantity to provide the rats with a diet containing 5% protein. Using the same basal ration as formerly (10) the test ration of ethanol-extracted

endosperm (Experiment 16) contained only 3.6% crude protein. This low percentage of protein may partly account for the rather high biological value of 70 established for this sample (Experiment 16). This value of 70, which is practically as high as the highest values obtained for corn germ reported in Table I, probably indicates that the residual proteins not extracted by the ethanol from the endosperm were of high quality. This may account in part for the high biological values obtained for these corn endosperm proteins, especially when compared to the biological value of the Iowa 939 waxy gluten used in Experiment 6. This result might have been predicted from the early work of Osborne and Mendel (6, 7) which demonstrated the superior value of the corn glutelins when compared to zein and corn gluten.

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COMPARISON OF CERTAIN CHEMICAL, PHYSICAL, AND BAKING PROPERTIES OF COMMERCIAL, BUHLER, AND HOBART-MILLED FLOURS [1]

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ABSTRACT

Commercial, Buhler, and Hobart flours were extracted from subsamples of each of seven commercial lots of wheat, representing the classes soft red winter, hard red winter, and hard red spring. The three types of millings were compared with respect to flour yield, loaf volume potentialities, mixing and oxidation requirements, cookie spread factor and top grain, and other physical and chemical properties including granulation, baking absorption, gas production, and crude fat, ash, and protein contents. The Hobart grinder, in conjunction with the wheat roll electrodes of a Tag-Heppenstall moisture meter, can be used as a micro-mill for extracting flours having physical, chemical, and baking properties that are comparable to those for Buhler and commercial-milled flours.

One of the needs in the development of simple methods for evaluating the quality of small samples of wheat was fulfilled by the work of Finney and Yamazaki (5) who describe a simple and rapid micro-milling procedure utilizing a Hobart grinder in conjunction with the wheat roll electrodes of a Tag-Heppenstall moisture meter. Their paper describes the techniques used in obtaining flours having ash and protein contents comparable to those obtained by the usual experimental milling procedures. Questions pertaining to the normalcy of Hobart-milled flours with respect to certain physical, baking, and other chemical properties were reserved for a separate investigation that is reported at this time.

Materials and Methods

Commercial, Buhler-experimental, and Hobart-micro-experimental flours were extracted from subsamples of each of seven commercial lots of wheat representing soft red winter, hard red winter, and hard red spring classes. The following milling companies supplied commercially milled flour and subsamples of the grain from which extracted.

[1] Manuscript received August 18, 1948. Presented at the Annual Meeting, May, 1947.
Cooperative investigations between the Division of Cereal Crops and Diseases, Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, U. S. Department of Agriculture, and the Departments of Agronomy and Milling Industry, Ohio and Kansas Agricultural Experiment Stations, respectively. Contribution No. 153, Department of Milling Industry.

[2] Hard Winter Wheat Quality Laboratory, Kansas Agricultural Experiment Station, Manhattan, Kansas.
[3] Federal Soft Wheat Laboratory, Ohio Agricultural Experiment Station, Wooster, Ohio.

	Class of Wheat
Mennel Milling Co., Fostoria, Ohio	SRW
J. C. Lysle Milling Co., Leavenworth, Kansas	SRW
Midland Flour Milling Co., Kansas City, Mo.	HRW
Kansas Milling Co., Wichita, Kansas	HRW
Montana Flour Mills Co., Great Falls, Montana	HRW
Montana Flour Mills Co., Great Falls, Montana	HRS
Bay State Milling Co., Winona, Minnesota	HRS

All commercial-milled flours were straight grade, excepting the Bay State HRS which was a short patent. All Buhler and Hobart flours were straight grade.

The Buhler millings were made as described by McCluggage, Anderson, and Larmour (6). The procedures and techniques for Hobart-milling, bread and cookie baking, and the determination of absorption, dough mixing requirement, and bromate requirement are described by Finney *et al.* (5, 3, 7, 2, and 4). Gas production, crude fat, ash, and protein content were determined according to Cereal Laboratory Methods (1). Granulation was determined by sifting 50 g. of flour for one hour in a Ro-Tap shaker equipped with 115, 150, 200, and 270 mesh per inch Tyler screens. Carmichael cleaners were placed beneath each screen. Preliminary studies indicated the necessity, particularly on the commercial SRW flours, of sifting for one hour and of brushing off each screen every 15 minutes.

Results

Flour Yield. Flour yields for the commercial, Buhler, and Hobart milling procedures are given in Table I. The higher yields for the commercial flours compared to those extracted on the Buhler and Hobart mills is due, undoubtedly, to the long system and large quantity of material milled. It should be pointed out that the Hobart grinder is not regarded as a mill for characterizing wheats as to milling properties and potentialities, although the possibilities in this direction are good when in the hands of an experienced operator. Its chief value is for extracting representative flour samples from quantities of grain too small to mill satisfactorily in the Buhler or Allis experimental mills. Research and quality evaluation studies can be carried out on these flours using micro methods. Even for tests ordinarily requiring larger amounts of flour, limited but valuable information can be obtained.

Flour Ash. Flour ash contents for the commercial, Buhler, and Hobart-milled flours are shown in Fig. 1. The ash values for the Hobart flours closely parallel, but are consistently higher than, those for the Buhler flours. The obvious lack of correlation between the commercial flour ash contents and those for the Buhler and Hobart experimental flours is not regarded as being inconsistent, particularly

in view of the lack of comparability of the commercial millings. For example, the hard red spring flour from Bay State Milling Company was a short patent. This flour together with the Montana hard red

TABLE I

MILLING, CHEMICAL, AND PHYSICAL DATA FOR COMMERCIAL, BUHLER, AND HOBART-MILLED FLOURS

Class and source	Coml.	Buhler	Hobart	Coml.	Buhler	Hobart
	FLOUR YIELD, %			FLOUR PROTEIN, %		
SRW —Mennel	69.4	62.0	63.6	7.9	8.0	8.1
SRW —Lysle	71.5	64.7	63.6	9.2	8.9	9.3
HRW—Montana	73.5	68.5	65.2	10.7	10.8	10.6
HRW—Midland	73.1	65.7	63.4	11.2	11.0	11.2
HRW—Kansas	73.5	66.0	63.9	11.0	10.9	11.3
HRS —Montana	72.2	64.2	63.5	13.1	13.6	14.4
HRS —Bay State	58.0[1]	63.3	64.7	12.1[1]	12.1	13.2
	CRUDE FAT, %			ABSORPTION (BREAD BAKING) %		
SRW —Mennel	1.1	0.9	1.2	51.0	50.5	50.5
SRW —Lysle	1.0	0.9	1.2	58.5	56.0	58.0
HRW—Montana	1.1	1.0	1.2	60.0	58.5	58.5
HRW—Midland	1.3	1.0	1.2	65.0	63.5	64.0
HRW—Kansas	1.1	0.9	1.0	67.5	64.5	65.0
HRS —Montana	1.2	1.0	1.5	63.5	63.0	64.0
HRS —Bay State	1.1[1]	1.0	1.4	64.5[1]	61.0	62.0
	MIXING REQUIREMENT, MIN.			BROMATE REQUIREMENT, MG.		
SRW —Mennel	2⅝	2⅝	2⅝	2	3	2
SRW —Lysle	3	2⅞	3	2	2	3
HRW—Montana	1¾	1¾	1⅝	6	6	6
HRW—Midland	2⅞	3	3¼	3	3	3
HRW—Kansas	3	2⅞	2⅞	4	4	4
HRS —Montana	3¼	3½	3¼	2	2	2
HRS —Bay State	3½[1]	3	3	2[1]	2	2

[1] Short patent—all others straight grade.

spring and the two soft red winter flours were lower in flour yield to varying degrees than the three hard red winter commercial-milled flours, thereby accounting for lower ash values.

Flour Protein and Crude Fat. Values for these tests are given in Table I. Crude fat indicates the relative amounts of wheat germ extracted by the three milling procedures. Both protein and crude

fat contents for the commercial, Buhler, and Hobart millings are in good agreement for the soft and hard red winter classes. For the hard red spring wheat flours extracted on the Hobart grinder, however, protein contents averaged about 1% higher and crude fat values about 0.4% higher than the corresponding values for commercial and Buhler millings. The extreme hardness of the two hard red spring wheats probably accounts for the relatively high protein and crude fat contents of the two corresponding Hobart-milled flours. These probably could be reduced by making minor changes in the milling procedure.

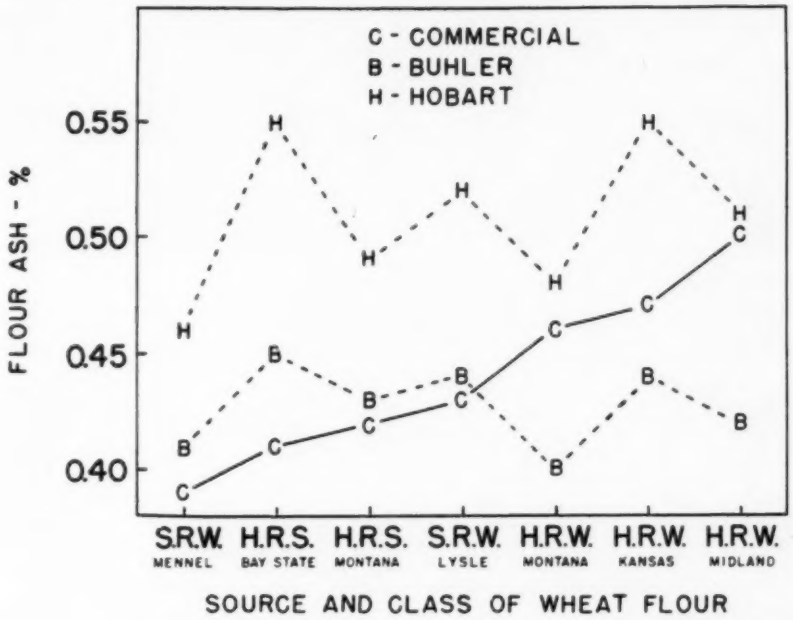

Fig. 1. Flour ash for commercial, Buhler, and Hobart-milled flours.

Granulation. Comparative granulation data obtained on the three millings representing each of the seven commercial wheats are given in Table II. The results are quite consistent in that a nearly negligible quantity of flour remains over the 115-mesh sieve for the commercial and Buhler flours in contrast to an average of 4.1% for the Hobart millings. Similarly, the overs of the 150-mesh sieve for the Hobart millings are considerably greater than for the two other millings. The greater amounts of overs of the 115- and 150-mesh sieves for the Hobart millings account, to a large extent, for the appreciably smaller amounts of material over the 200- and 270-mesh sieves compared to the commercial and Buhler millings. These granulation differences undoubtedly are accounted for largely by the differences between the extrac-

tion systems and flour sieves used for the three mills. In contrast to the long commercial system, the Buhler millings were made with three breaks and three reductions, and those on the Hobart with only one break and two reductions. Where the equivalent of a 9XX flour cloth was used in the Hobart procedure, 10XX flour cloths were used on the Buhler, and 10XX to 11XX cloths were used for the commercial

TABLE II

GRANULATION OF COMMERCIAL, BUHLER, AND HOBART-MILLED FLOURS

Sample	Overs 115	Overs 150	Overs 200	Overs 270	Throughs 270
	%	%	%	%	%
Mennel—S.R.W.					
Coml.	0.0	0.2	10.8	19.4	62.8
Hobart	1.0	10.0	15.2	8.4	60.0
Buhler	0.2	2.0	27.0	14.8	52.4
Lysle—S.R.W.					
Coml.	0.2	2.0	23.2	18.6	51.0
Hobart	2.8	15.6	19.4	11.0	45.4
Buhler	0.0	2.8	32.6	16.6	45.0
Bay State—H.R.S.					
Coml.	1.4	11.6	29.4	19.4	34.6
Hobart	4.4	20.0	24.6	14.0	33.0
Buhler	0.0	6.2	41.6	17.2	31.4
Montana—H.R.S.					
Coml.	0.2	4.2	27.4	22.2	43.0
Hobart	4.4	19.2	23.6	12.2	36.2
Buhler	0.0	5.4	40.8	18.4	31.8
Montana—H.R.W.					
Coml.	0.2	5.8	32.8	19.8	39.6
Hobart	4.8	19.6	22.6	11.8	36.6
Buhler	0.0	3.8	39.0	19.8	33.0
Midland—H.R.W.					
Coml.	0.2	1.6	22.2	23.2	49.6
Hobart	5.6	20.6	23.0	9.8	38.0
Buhler	0.0	3.2	35.8	20.2	37.0
Kansas—H.R.W.					
Coml.	0.2	3.8	26.0	20.0	47.6
Hobart	5.8	20.4	23.0	13.2	36.2
Buhler	0.0	4.4	38.2	19.0	35.4

millings. It is pertinent to note, however, that the throughs of the 270 for the Hobart flours are consistently greater than those for the Buhler millings by an average of 2.8%. Considering the differences in the three mills, the average granulations for the three types of millings are surprisingly similar.

4th Hour Gas Production. Gassing data for the seven soft and hard wheat flours extracted by each of the three milling procedures are

shown graphically in Fig. 2. The results for the Buhler and Hobart millings are nearly identical and obviously are highly correlated with those for the commercial flours. These data are in accord with the granulation data, since they indicate that the Buhler and Hobart millings have about the same average flour particle size, and are appreciably coarser than the commercial flours.

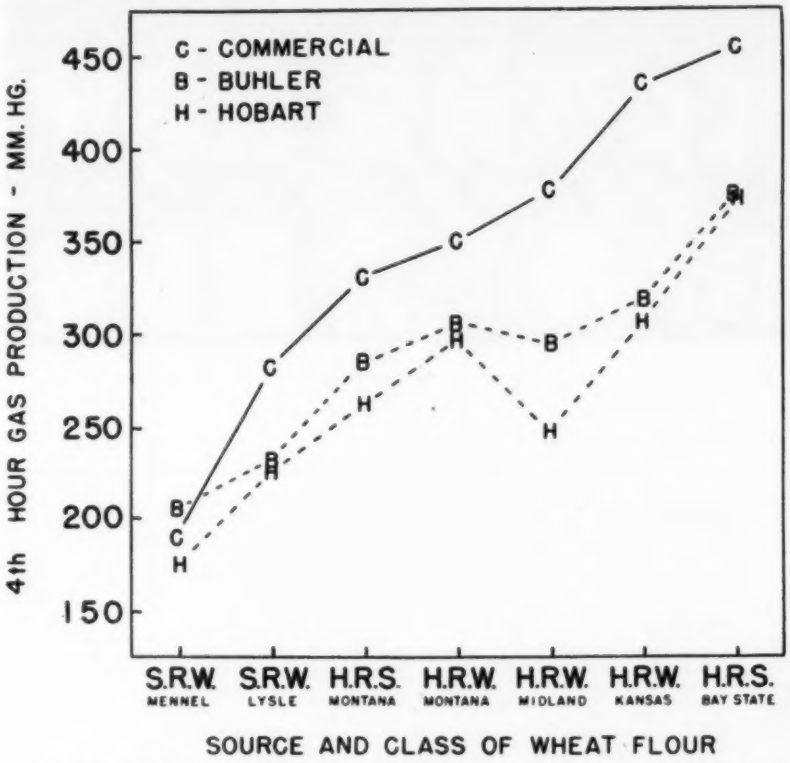

Fig. 2. Fourth hour gas production for commercial, Buhler, and Hobart-milled flours.

Absorption, Mixing, and Bromate Requirements. A study of the relation of milling procedure or technique to absorption, mixing, and bromate requirements is warranted because of their importance in baking technology and the production of good bread. The data for baking absorption, dough mixing time, and potassium bromate requirement are given in Table I. It is apparent from an examination of these data that, for a given test, a very high correlation exists between the results for any two milling procedures. As a matter of fact, the variation due to milling method is practically negligible for mixing and bromate requirements. The somewhat higher absorption, in general, for the commercially extracted flours is probably due to their finer particle size.

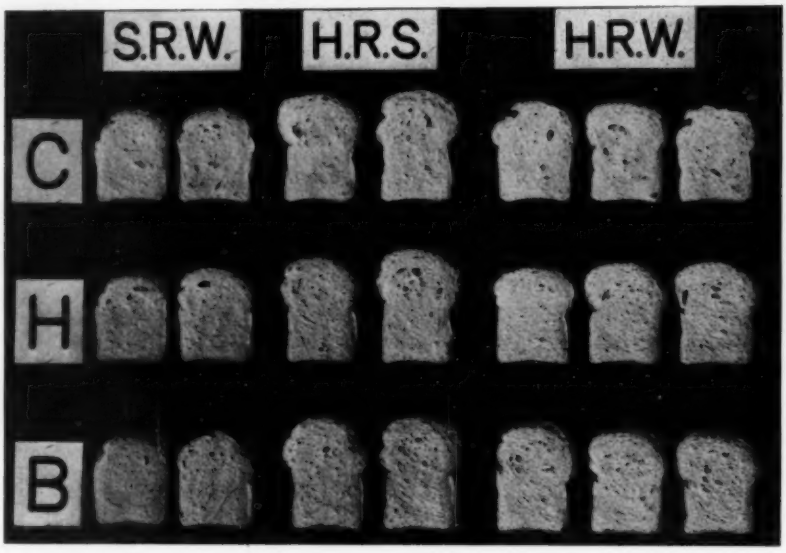

Fig. 3. Loaves of bread baked from commercial, Buhler, and Hobart-milled flours. Source of flour (left to right): Mennel, Lysle, Bay State, Montana, Montana, Midland, Kansas.

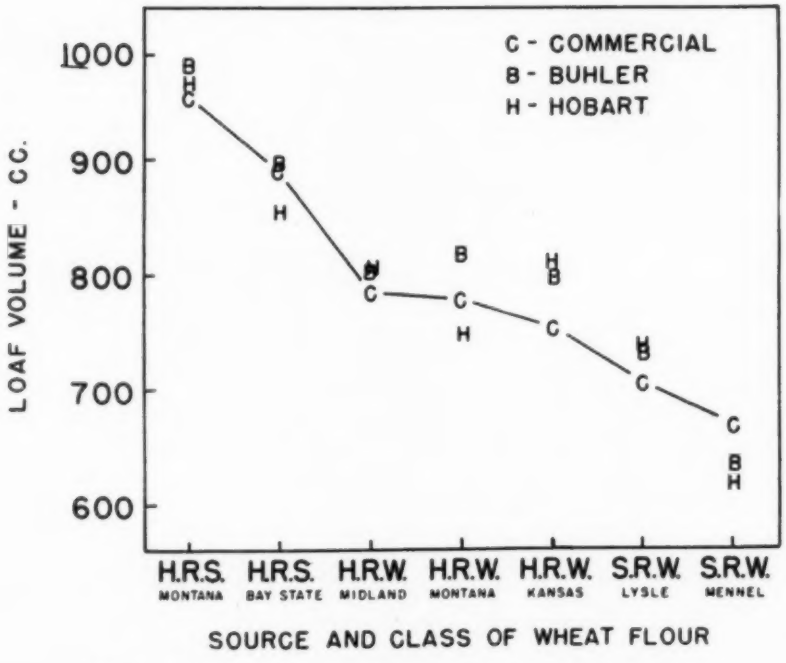

Fig. 4. Loaf volume for commercial, Buhler, and Hobart-milled flours.

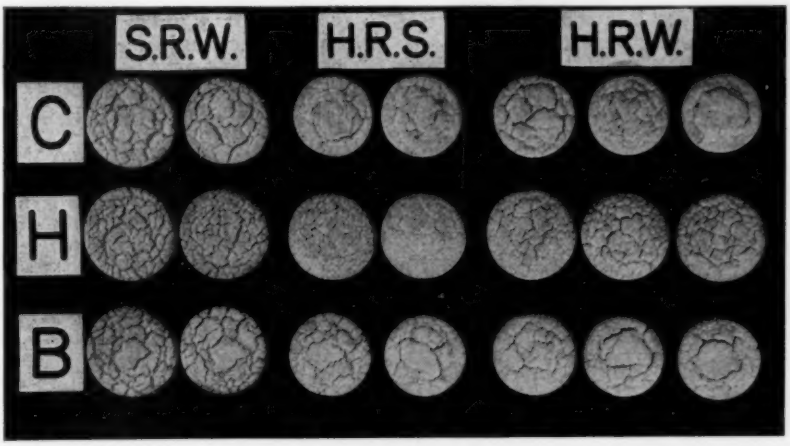

Fig. 5. Sugar snap cookies baked from commercial, Buhler, and Hobart-milled flours. Source of flour (left to right): Mennel, Lysle, Bay State, Montana, Montana, Midland, Kansas.

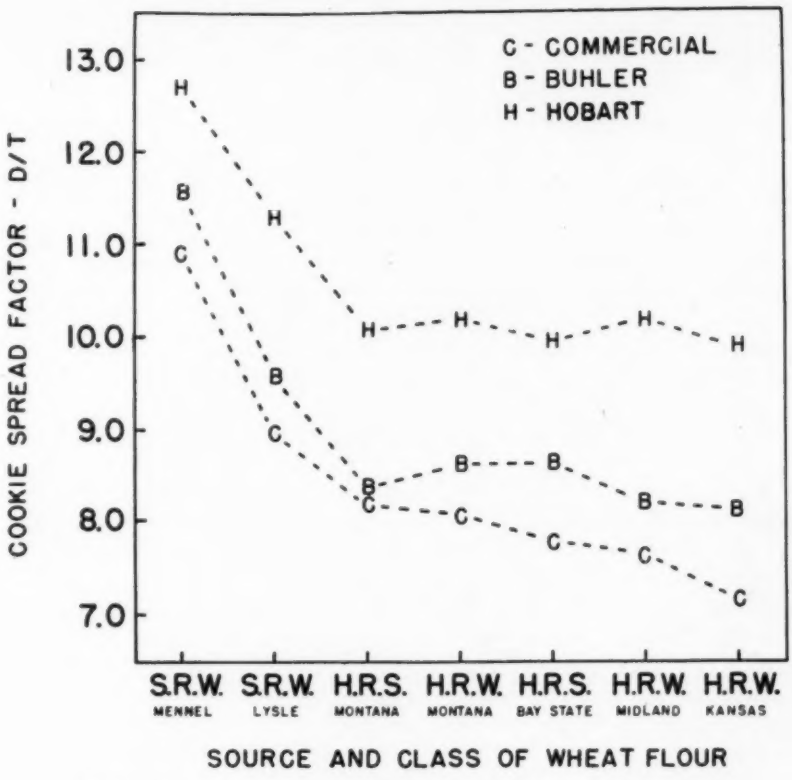

Fig. 6. Cookie spread factor (diameter/thickness) for commercial, Buhler, and Hobart-milled flours.

Loaf Volume of Bread. Internal characteristics and relative size of loaves of bread representing the seven different wheats and three mills are shown in Fig. 3. Loaf volumes are shown graphically in Fig. 4. These figures show that flours extracted on the Hobart grinder will yield bread having crumb grains and loaf volumes equal or very similar to those for flours extracted on the Buhler and commercial mills.

Cookie Spread Factor (D/T). Top grains and relative diameters for cookies baked from the 21 flours used throughout these studies are shown in Fig. 5. Cookie spread factors (diameter/thickness) are presented graphically in Fig. 6.

The well-broken top grain and greater cookie spread factor produced from the Hobart flours indicate superior cookie baking quality. While this superiority of the Hobart flour cookies may be partially attributed to differences in particle size distribution, nevertheless the spread factors (Fig. 6) for the Hobart-milled flours are highly correlated with those for the commercial and Buhler-milled flours. This is evident since the two soft winter flours are ranked in the same order and above the hard winter and spring flours for all three milling procedures.

Acknowledgment

Credit is due Gerald D. Miller, Department of Milling Industry, Kansas State College, for making the flour ash, protein, crude fat, and gas production determinations.

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